United States Environmental Protection Agency

Region 10, 1200 Sixth Avenue, Seattle WA 98101

QUALITY ASSURANCE

PROJECT PLAN

ASSESSMENT OF CHEMICAL CONTAMINANTS IN FISH CONSUMED BY FOUR NATIVE AMERICAN TRIBES IN THE COLUMBIA RIVER BASIN

Revision 6.0

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Prepared By

U.S. Environmental Protection Agency (EPA)
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1.0 PROJECT DESCRIPTION

The U.S. Environmental Protection Agency (EPA) has initiated a study to assess chemical contaminant exposure from consumption of Columbia River fish by four Native American Tribes (Nez Perce, Warm Springs, Umatilla, and Yakama). These tribes are also referred to as Columbia River Treaty tribes. The first phase of this study was completed in October of 1994 by the Columbia River Inter-Tribal Fish Commission (CRITFC).

This current phase of the study (referred to as Phase II), will consist of evaluating tissue contaminant data representing resident and anadromous fish species that are caught by tribal fisheries in the Columbia River Basin and consumed by tribal members. This Quality Assurance Project Plan (QAPP) is the overall planning document for Phase II of the study.

The information from both phases of this exposure study will then be used to assess the potential health impacts to the Columbia River Treaty Tribes from consuming contaminants in Columbia River fish.

1.1 HISTORICAL OVERVIEW

Several studies have shown that elevated levels of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzo-p-furans (PCDDs/PCDFs) are present in the biota of several areas of the Columbia River Basin. Measurements of the levels of PCDDs/PCDFs at pulp and paper mills lead to the conclusion that water discharges from these mills were the primary source of these contaminants. As a result, in 1991, Region 10 EPA established a Total Maximum Daily Load (TMDL) for 2,3,7,8-TCDD (2,3,7,8-tetrachlorodibenzo-p-dioxin) which provides a framework to allocate the permissible 2,3,7,8-TCDD loading to the Columbia River Basin. In response to this TMDL, pulp and paper mills in the Columbia River Basin have been required, through permits issued under the National Pollutant Discharge Elimination System (NPDES), to modify their processes to achieve non-detectable levels of 2,3,7,8-TCDD in their wastewater discharges. Despite the expected decrease in discharges of this pollutant from pulp and paper mills, there is still concern about consumption of biota contaminated with PCDDs/PCDFs from the Columbia River. This is because exposures to extremely low levels of the PCDDs/PCDFs may result in adverse impacts on human health and because this class of compounds are highly bioaccumulative and persistent in the environment.

Studies in the Columbia River Basin have also shown that there are elevated levels of other contaminants of concern in the biota and sediments, including polychlorinated biphenyls (PCBs), and chlorinated pesticides, and inorganics. For example, as shown in a study done by the United States Geological Survey (7), DDT and its breakdown products, DDE and DDD, are still elevated in the water, sediment and fish in the Yakima River Basin (which is a part of the Columbia River Basin) despite the fact that two decades have passed since the production and distribution of DDT

was banned in the U.S. This study also concluded that fish in the Yakima Basin have among the highest concentrations of DDT in the Nation.

The fishery resource in the Columbia River Basin is not only a major food source for tribal members but it is also an integral part of the tribes' cultural, economic, and spiritual well-being. Because fish are consumed for both subsistence and ceremonial purposes, there has been concern that tribal members may be highly exposed to contaminants in fish because they consume large amounts of fish and eat fish body parts (e.g., fish eggs) that tend to accumulate fat-soluble toxins, like PCDDs/PCDFs, PCBs, and chlorinated pesticides. The Columbia River Treaty tribes have questioned the adequacy of the 6.5 gram per day fish ingestion rate used by the EPA to develop the TMDL for 2,3,7,8-TCDD in the Columbia River Basin. This value of 6.5 grams per day, which is also used by EPA in developing its national ambient water quality criteria for protection of human health from consumption of aquatic life, is the estimated average national fish consumption rate based upon a U.S. national diet survey.

Because of the elevated levels of contaminants in the Columbia River Basin and because of the importance of fish to the tribes in the Basin, the U.S. EPA initiated a two-phase exposure study to examine the role of fish consumption as an exposure route for waterborne contaminants among individuals of four of the Columbia River tribes.

In Phase I of this exposure study, the U.S. EPA entered into a Cooperative Agreement with the Columbia River Inter-Tribal Fish Commission (CRITFC) in 1990 to formally conduct a fish consumption survey of the four tribes represented by CRITFC - the Umatilla, Nez Perce, Yakama, and Warm Springs. This consumption study, published by CRITFC in October of 1994 (1), documented the types and amounts of fish eaten by tribal members as well as the fish parts consumed and food preparation methods used. The average fish consumption rate of adult tribal members (combining both fish consumers and non-fish consumers) was 58.7 grams per day. This value is about 9 times higher than the national average fish consumption rate (6.5 grams per day) used by the EPA. The 95th percentile of consumption for adult tribal members (combining both fish consumers and non-fish consumers) was approximately 170 grams per day. The location and frequency of use of tribal fishing sites in the Columbia River Basin, which is the source of about 90% of the fish consumed by tribal members, were also documented in the survey.

Phase II of this exposure study will use the information from the consumption study and from existing data on the levels of contaminants in Columbia River fish to design and implement a sampling program to collect tissue contaminant data from resident and anadromous fish species consumed by tribal members. The QAPP detailed in this document will be used for the sampling and analysis program in this second phase. The data from the first (fish consumption survey) and second (tissue contaminant data) phases of this exposure study will provide information that can be used to estimate the potential health impacts from consumption of Columbia River fish for these four tribes.

1.2 SURVEY OBJECTIVES

Phase II is being designed and implemented by EPA with input from representatives of CRITFC and its four member tribes, the U.S. Fish and Wildlife Service, the U.S. Geological Survey, and the Washington and Oregon State health and environmental agencies.

Prior to the development of this QAPP, a preliminary scoping document (2) was prepared for EPA by Tetra Tech, an EPA contractor, using data from: (a) the CRITFC fish consumption study; (b) personal communications with tribal fishery managers and tribal fishers; and © a data base compiled by Tetra Tech which summarized existing contaminant data on biota in the Columbia River Basin. This scoping document included a discussion of study objectives and a preliminary study design. At a design conference held in Portland, Oregon, on October 19-20, 1994, and attended by representatives of the organizations listed above, changes to the preliminary scoping document were recommended. The final scoping document, Assessment of Chemical Contaminants in Fish Consumed by Four Native American Tribes in the Columbia River Basin - Final Draft Study Design, was completed on December 2, 1994 (3) (referred to as "draft study design document" from here on).

The objectives for Phase II, as discussed in the draft study design document, are to:

- Measure fish contaminant levels for species and fishing locations being utilized by CRITFC member tribes to provide, in conjunction with the CRITFC fish consumption report, an assessment of fish consumption as an exposure route for waterborne chemical toxics among individuals of these tribes.

- Use the information derived from the exposure assessment to estimate potential health risks to fish consumers in the four CRITFC member tribes.

The discussion surrounding these two objectives is discussed in more detail in the draft study design document.

1.3 DOCUMENT PURPOSE AND SCOPE

This document provides technical and procedural guidance and requirements to ensure that a well-planned scientific investigation is conducted, and that the field measurements and analytical data obtained serve the project objectives described above. The content and structure of this QAPP are based upon requirements and guidelines in Quality Management Program Plan For Region 10, EPA Region 10, Seattle, WA, RQMP-001/92, January 23, 1993, which requires the use of Interim Guidelines and Specifications For Preparing Quality Assurance Project Plans, QAMS-005/80, December 29, 1980, for the preparation of QAPPs involving sampling and analysis projects in EPA Region 10. Specifications for data quality are presented in Section 3.0. Preparation of this QAPP helps the project manager focus on the factors affecting data quality during the planning stage of the project. The completed plan defines field and laboratory procedures, and facilitates project implementation and communication among field, laboratory, and management staff.

Communication is extremely important for this project because of the number of different organizations and individuals involved as shown in Figure 1. The Project Manager at EPA Region 10 in Seattle, Washington, is also the Work Assignment Manager (WAM) for the EPA Contractor, Tetra Tech. CRITFC's Water Quality Manager is the Tribal Manager for this project.

The EPA Field Operations Manager (FOM) in Region 10 will coordinate a field crew of EPA and tribal staff to collect fish samples. Fish samples to be analyzed as whole fish will be sent to a laboratory which is a subcontractor to the EPA Contractor, Tetra Tech. For those fish in which fillets and/or eggs are to be measured, fish will first be filleted and the eggs collected by the EPA and other field crew members and these fillets and eggs will then be sent to the subcontract laboratory. The subcontract laboratory will be responsible for homogenizing all of the fish and egg samples and for preparing sample aliquots for all analyses. The subcontract laboratory will also analyze the fish and egg samples for chlorinated dioxins and furans and toxic, dioxin-like, PCBs (often referred to as coplanar PCBs).

The subcontract laboratory will also send samples of the homogenized samples to the EPA Region 10 Laboratory for analysis (pesticides/PCBs, semivolatiles including polyaromatic hydrocarbons, and inorganics) and for archiving. Some of these archived samples may be used for analysis of radionuclides at another laboratory if resources become available.

Data quality review of all analytical data will be performed by EPA Region 10. Analytical data and documentation for toxic, dioxin-like PCBs and chlorinated dioxins and furans generated by the subcontract laboratory will be sent to the Contractor, Tetra Tech. Tetra Tech will then send copies of these data and documentation to EPA, Region 10, where a validation of the data will be conducted by the EPA Project QA Manager. Validation of data from the EPA Region 10 Laboratory (pesticides/PCBs, semi-volatiles and inorganics) will be conducted by the Region 10 Laboratory. The EPA Project QA Manager will also perform a data quality review of radionuclide data if resources are found for these analyses. All validated data will then be sent to Tetra Tech where it will be compiled into a summary data report and entered into the Columbia River contaminant database which was previously developed for EPA by Tetra Tech.

This QAPP details procedures for field sampling, filleting and homogenization of fish, and chemical analyses. In addition, protocols for documentation, labeling, handling, chain of custody, storage and shipping, and analytical QA procedures are discussed. Field and laboratory procedures are described in Sections 4.0 through 9.0. Sections on data validation and review (10.0); quality control procedures (9.0); preventive maintenance (12.0); data assessment and reporting(14.0); and corrective actions (13.0) provide sufficient detail to direct activities of project participants and provide interested readers with an understanding of how analytical data will be used in project decision-making processes.

A Cooperative Agreement (see Attachment 1) has been developed between CRITFC and U.S. EPA, Region 10. The purpose of this Agreement is to set forth the relationship and nature of cooperation between CRITFC and EPA in all aspects of the Phase II study including, but not limited to, sample collection, tissue analysis, data assessment, and data release. The work done in this QAPP will be done in cooperation with CRITFC as written in the Cooperative Agreement.

A project schedule of major milestones for sample collection, data analysis, validation and assessment of data, and final project report preparation can not be accurately specified for the project due to major variables such as multi source funding and variations in fish populations. Table 7 provides a schedule of sampling activities for the project. Tables 7 and 8 from the previous revision of the QAPP (Revision 5.1, June 17, 1996) are provided in Attachments 19 and 20 to this QAPP. The project sampling schedule will need to be flexible due to variations in fish populations. A Sample Alteration Form (see Attachment 17) will be prepared and approved when the sampling schedule requires changing. Reservations for laboratory measurements will be made by the FOM with the EPA Manchester Laboratory 45 days prior to shipment of samples. Validation of project data will be completed within 60 days of receipt of laboratory reports. Final data assessment and submission of the draft final report for the project will be completed on or before June 1, 1998.

2.0 PROJECT ORGANIZATION

The managerial organization of the project is shown in Figure 2. Project Managers have the following assigned responsibilities:

- **Project Manager: Pat Cirone**

  USEPA, Region 10
  (206) 553-1597
  fax: (206) 553-0119

  Pat Cirone will be responsible for the overall quality of data and project activities for EPA Region 10. She will be responsible for ensuring that Region 10 project staff clearly understand their responsibilities and authority on the project. The Regional Project Manager consults with the Project Leader and approves all deviations from the QAPP. The Regional Project Manager reviews all audit reports and ensures that corrective actions or non-conformances are taken in a timely and appropriate manner. The Project Manager is responsible for ensuring that the QAPP is adequately reviewed prior to implementation of the project.

  Pat Cirone is also the Work Assignment Manager (WAM) for the contract with Tetra Tech/Redmond. As the Work Assignment Manager for Tetra Tech, she is responsible for ensuring that Tetra Tech and all subcontractors of Tetra Tech, implement the specifications and requirements of the QAPP. Tetra Tech's role in the project is to carry out the requirements of the Work Assignment which is issued and managed by Pat Cirone.

- **Regional Project Leader: Dana Davoli**

  USEPA, Region 10
  (206) 553-2135
  fax: (206) 553-0119

  The Regional Project Leader reports directly to the Region 10 Project Manager. All other Regional Project staff report directly to the Regional Project Leader. The Regional Project Leader is responsible to the Regional Project Manager for implementing and carrying out the requirements of the QAPP for Region 10. All information concerning project activities is transmitted by Region 10 staff through the Regional Project Leader to the Regional Project Manager.

! **Field Operations Manager: David Terpening**

USEPA, Region 10
(206) 553-6905
fax: (206) 553-0119

The FOM is responsible for planning and implementing field activities, including fish collection, fish filleting and egg collection, and shipment of samples to the contract lab. In order to carry-out these responsibilities, the FOM will communicate frequently and periodically with the Regional Project Leader and the Project Manager concerning field activities. The FOM will report to the Project Leader.

! **Regional Lab Project Coordinator: Peggy Knight**

USEPA, Region 10
(360) 871-8713
fax: (360) 871-8747

The Regional Laboratory Project Coordinator will be responsible for coordination and oversight of the EPA Region 10 Laboratory's work on the Project. Peggy Knight will monitor laboratory activities and coordinate communications of laboratory activities and laboratory reports to the Regional Project Leader. The Regional Lab Coordinator will report to the Project Leader.

! **Project QA Manager: Robert G. Melton**

USEPA, Region 10
(206) 553-2147
fax: (206) 553-8210

The Project QA Manager is responsible for implementation of all QA requirements of the QAPP. He will be the primary data quality reviewer of the analytical results (PCDD/PCDF congeners and dioxin-like PCBs) from the subcontract laboratory and, if resources become available, from the laboratory(s) conducting radionuclide analyses. He oversees laboratory performance and quality control requirements of the QAPP. The Project QA Manager is responsible for documenting to the Project Leader and Project Manager that corrective actions have been implemented. The Project QA Manager must review and approve the QAPP before the QAPP can be implemented. The Project QA Manager reports on routine project matters to the Project Leader.

! **Regional QA Manager: Barry Towns**

USEPA, Region 10
(206) 553-1675
fax: (206) 553-0119

The Region 10 Quality Assurance Unit (QAU)is responsible for to review and concur on the approval/disapproval of QAPPs required by EPA Order 5360.1 (see Region 10's Quality Management Plan of 1992). Final approval/disapproval of this QAPP lies with project management personnel. In executing his QA and oversight responsibilities, the Regional QA Officer reports to the Regional Administrator.

! **Columbia River Inter-Tribal Fish Commission (CRITFC):**

CRITFC
(503) 238-0667
fax: (503) 235-4228

As discussed above, a Cooperative Agreement has been developed between CRITFC and U.S. EPA, Region 10 which sets forth the relationship and nature of cooperation between CRITFC and EPA in all aspects of the Phase II study.

Figure 1. Flow Diagram of Project Tasks

3.0 DATA QUALITY OBJECTIVES

The overall QA objective for analytical data is to ensure that data of known and acceptable quality are produced so that potential health risks to fish consumers in the four CRITFC member tribes can be estimated. Data quality objectives (DQOs) for the project are discussed below, in Table 1, in the attachments to the QAPP, and in other sections of the QAPP. Project DQOs include:

- 1. The selection of the appropriate chemical target compounds to be measured and the appropriate quantitation limits for these compounds, and,

- 2. Analytical objectives as defined by measurement of PRECISION, ACCURACY, REPRESENTATIVENESS, COMPLETENESS, and COMPARABILITY of quality assurance samples such as field duplicate samples, performance evaluation samples, and laboratory quality control samples. These QA samples will be used to evaluate project data to determine if data meets the specified DQOs of the QAPP.

3.1 SELECTION OF TARGET COMPOUNDS AND DETECTION LIMITS

As discussed in Section 1.2, the objectives for Phase II are to measure fish contaminant levels for species caught at fishing locations being utilized by CRITFC tribal members. These data will then be used to provide information on potential exposures and health impacts from waterborne chemical toxics for these tribes.

The selection of target compounds and the risk-based detection limit goals were determined in the draft study design document prepared for this project by Tetra Tech (3). In this document, target analytes were selected by considering guidance provided by the U.S. EPA (4) and by performing a health risk-based screening analysis of tissue contaminant data collected within the Columbia River Basin during the last ten years (1984-1994).

Screening for carcinogenic effects was performed for a 70 kg adult using a target cancer risk of 1×10^{-6}. Screening for non-cancer effects was performed for a 14.5 kg child using a target hazard quotient of 0.1. Fish consumption rates assumed for adults and children were 194 and 81 g/day, respectively, which correspond to the cumulative 97th percentile consumption rate reported in CRITFC (1). For chemicals that had both slope factors for estimating carcinogenic risk and reference doses for estimating non-carcinogenic impacts, separate tissue screening concentrations (STCs) were calculated and the lower of the two values was used for the screening analysis. These STCs were then compared to the tissue contaminant data collected in the Columbia River Basin in the past ten years.

Only a small number of chemicals from this tissue contaminant data did not exceed the STCs. Chemicals that exceeded the STCs included dioxins/furans, PCBs, organochlorine and organophosphorus pesticides, PAHs and other semivolatiles, trace metal and radionuclides. Based on this risk screening analysis, a decision was made to measure for the contaminant classes listed in Table 1. Table 2, and Tables 4 through 6 provide a listing of individual contaminants in each of these classes.

After the draft study design document was completed, an analytical method (Method 1668) became available for measuring toxic dioxin-like PCBs. These compounds were added to the list of analytes and are shown in Table 3.

The STCs calculated in the study design document were selected as the risk-based detection limit goals for this project with the following exception. In the design document, the fish consumption rates used to calculate the STCs were the 97th percentile from the CRITFC study. Because use of the 95th percentile is more in line with EPA guidance, the STCs in the design document were recalculated using the 95th percentile consumption rates. Table 2 and Tables 4 through 6 contain the risk-based detection limit goals (formally the STCs)(shown in the tables as the "risk levels") calculated using the 95th percentile fish consumption rates. The quantitation limits that will be achieved in this project are also included in these tables. Analytical detection limits for the toxic, dioxin like PCBs are shown in Table 3.

Radionuclides are not included in Table 6 (inorganics) due to lack of resources to pay for analytical measurements at this time. However, it was agreed at the scoping meeting that EPA would attempt to find resources for these analyses.

As shown in Tables 2-6, several chemicals have detection limits that are above the risk level goals that were calculated. For this project, the analytical methods being used were chosen to provide detection or quantitation limits which are as low as possible given available analytical methods and resources.

3.2 MEASUREMENT OBJECTIVES

The following objectives are measurement goals for this project:

3.2.1 Precision

Precision is the measurement of agreement among repetitive measurements of the same sample. Precision will be evaluated in two ways:

(1) The relative percent difference (RPD) between matrix spike/matrix spike duplicate (MS/MSD) samples will be calculated. As shown in Table 1, MS/MSD measurements will be made at a frequency of one per twenty samples/composites. Since a total of 122 fish samples are expected to be measured in this project, this results in a total of approximately 7 MS/MSD samples for each analytical group.

(2) The relative percent difference (RPD) between field duplicate samples will be calculated. As shown in Table 7, for one composite sample of steelhead and one of spring chinook, separate composites of fillets will be prepared from each side of the fish. The comparison of the analytical results from both sides will serve as field duplicates. In addition, two blind duplicate field samples will be selected by the Project Manager for complete target compound analysis.

For field duplicate samples and for matrix spiked and matrix spiked-duplicate samples, precision will be measured as Relative Percent Difference (RPD).

$$RPD = \frac{ABS(R1 - R2)}{((R1 + R2)/2)} \times 100$$

R1 = Recovery for MS or duplicate 1, R2 = Recovery for MSD or duplicate 2

Precision required for the analysis of project MS/MSD samples is specified in Table 1. Precision required for the analysis of field duplicates (consisting of the opposite fillets of the same fish) shall be less than 40 relative percent difference.

3.2.2 Accuracy

Accuracy is the degree of agreement of an experimental measurement with an accepted standard reference. Accuracy will be evaluated by calculating the percent recovery (%R) of target analytes or isotope-labeled target compounds in spiked samples, and by the measurement of known target compounds in Performance Evaluation (PE) tissue samples.

$$\%\ Recovery = \frac{SQ - NQ}{S} \times 100$$

SQ = quantity found in spiked sample,
NQ = quantity found in native (unspiked) sample,
S = quantity of spike added to native sample

The accuracy requirements for MS/MSD samples for each measurement method are presented in Table 1. As discussed above, MS/MSD samples will be measured at a frequency of 1:20 for a total of 7 per analytical group.

As shown in Table 1, six Performance Evaluation (PE) samples (PE samples EDF-2524, EDF-2525, and EDF-2526) will be measured for chlorinated dioxins/furans and for the toxic, dioxin-like, PCBs. Accuracy requirements of acceptable recovery ranges for these PE samples have been documented by Cambridge Isotope Laboratories. These acceptable accuracy recovery ranges will be required by the laboratory which measures PCDDs/PCDFs and toxic, dioxin-like, PCBs. Blind PE samples for PCDDs/PCDFs and toxic, dioxin-like, PCBs measurements will not be used in this study because none are available in one kilogram quantities. However, two blind duplicate field samples will be selected by the Project Manager for complete target compound analysis.

3.2.3 Representativeness

Representativeness is the degree to which data from the project accurately represents a particular characteristic of the environmental matrix which is being tested. For example, representativeness is the degree to which data accurately and precisely represents a characteristic of a population, a matrix, a natural variation at a sampling location, or an environmental condition. Acceptable representativeness is achieved through adequate sampling program design and QAPP design. Goals for representativeness are primarily met by ensuring that, given available resources, sampling locations are properly selected and that a sufficient number of tissue types and fish species are collected. Sections 4 through 8 of the QAPP specify procedures which will be used to ensure that samples are representative of Columbia River basin fish which are consumed by the Columbia River Treaty tribes.

3.2.4 Completeness

Completeness is the percentage of valid results obtained as compared to the total number of samples taken for a parameter. Completeness requirements for this project are presented in Table 1.

$$\% \text{ Completeness} = \frac{\text{\# of valid results}}{\text{\# of samples taken}}$$

Figure 2. Project Organization

3.2.5 Comparability

Comparability is a qualitative characteristic expressing the confidence with which one data set can be compared with other data sets. In this regard, measurements of PCDDs/PCDFs and toxic, dioxin-like, PCBs from this project may not be comparable with PCDD/PCDF and toxic, dioxin-like, PCB data measurements from previous projects because new and improved state-of-the-art methods such as Methods 1613B and 1668 are used in this project to measure samples. In addition, data from previous projects have not always been validated and qualified by a chemist to determine data quality and data useability. Therefore, a comparability goal for the measurement of PCDDs/PCDFs and non-coplanar PCBs for this project cannot be set. By contrast, project data for the measurement of metals, pesticides/PCBs and semi-volatile organics should be more comparable to previous data from the analysis of Columbia River basin fish. A comparability goal of 70% is set for these non-PCDD/PCDF and non-coplanar PCB data.

The QA data quality objectives outlined above, will be evaluated in conjunction with the data validation process, and will be documented in the Final Summary Data Report for the project.

3.3 OTHER DATA QUALITY OBJECTIVES

In addition to the specific measurement objectives discussed above, Section 9.4 of the QAPP specifies that all quality control requirements of each method which is referenced in Table 1 shall be obtained and reported by each analytical laboratory. These laboratory QC measurements include the use of surrogate compounds, internal standards, recovery standards, matrix spike compounds, isotope dilution labeled internal standards, instrument calibrations, and method blanks.

As shown in Table 7 and discussed in Section 4.3, for all species except sturgeon, three composite samples will be collected at each sampling site for each species. These composites will be composed of different individual fish of the same species at a location close to the specified sampling station. The QAPP does not have a data quality objective for expected precision of these three composite samples of the same species. As discussed in Section 4.3, EPA guidance regarding numbers of fish per composite and length of fish will be followed if possible. However, these goals may not be possible if there is there is difficulty in catching fish. Relative Standard Deviations (RSDs) between the three composite samples collected at each sampling station will be calculated after analyses are completed.

An additional very important data quality objective of the project is to obtain validated PCDD/PCDF data which is free of expected chlorinated chemical interferences to the measurement of PCDD/PCDF target compounds, such as Polychlorinated Diphenyl Ether (PCDPE) interferences. Therefore, one of the additional primary data quality objectives in this QAPP is for the subcontract laboratory to remove chemical interferences to the measurement of 2,3,7,8-TCDF, which is the PCDD/PCDF isomer which has historically been found in the highest

concentrations in fish tissue in the Columbia River system. Previous data from the Columbia River system has often been contaminated with PCDPE chemical interferences.

Table 1. Sampling and Measurement Objectives and Requirements For the Project

Analytical Group	List of Target Com-pounds	Tentative Number of Field Samples[1,6]	Number of QA Samples: PE[2], MS/MSD[2] Dups[1]	Matrix	Method	Accuracy[3]	Precision[4] (RPD)	Complete-ness	Preser-vation	Containers (Field/Lab)	Holding Time For Project Samples
PCDDs/ PCDFs/ % Lipids	Table 2	251	6 PE's 8 Dups	Fish Tissue	1613B + SOW	70 to 140%	40%	90%	-20°C	Al foil/ 2x2ozWM	1 yr. (sample) 40 days (extract)
Toxic, Dioxin-Like, PCBs	Table 3	251	6 PE's 8 Dups	Fish Tissue	1668	70 to 140%	40%	90%	-20°C	Al foil/ 2x2ozWM	1 yr. (sample) 40 days (extract)
Chlorinated Pesticides/ Aroclors	Table 11	251	12 MS/MSDs 8 Dups	Fish Tissue	8081 Florisil/Acetonitrile Partitioning/Florisil	30-150%	50%	90%	-20°C	Al foil/ 2x2ozWM	1 yr. (sample) 40 days (extract)
AED/ Pesticides	Table 12	60 to 120	6 MS/MSDs 4 Dups	Fish Tissue	8085 Acetonitrile Partitioning	30-150%	50%	90%	-20°C	Al foil/ 2x2ozWM	1 yr. (sample) 40 days (extract)
Neutral Semivol.	Table 4	251	12 MS/MSDs 8 Dups	Fish Tissue	8270/ GPC/SG	10-150%	50%	90%	-20°C	Al foil/ 2x2ozWM	1 yr. (sample) 40 days (extract)
PAHs	Table 4	251	12 MS/MSDs 8 Dups	Fish Tissue	8270/SIM GPC/SG	30-140%	50%	90%	-20°C	Al foil/ use SV ext	1 yr. (sample) 40 days (extract)
Chlorinated Phenolics	Table 5	251	12 MS/MSDs 8 Dups	Fish Tissue	1653 Modified GPC/Acetylation	20-150%	50%	90%	-20°C	Al foil/ 2x2ozWM	1 yr. (sample) 40 days (extract)
Metals	Table 6	251	8 Dups	Fish Tissue	200.3 & 200.8[5]	60-140%	30%	90%	-20°C	Al foil/ 2x2ozWM	2 yrs.
Mercury	Table 6	251	8 Dups	Fish Tissue	251.6[5], Rev. 2.3	60-140%	35%	90%	-20°C	Al foil/ use ICP WM	86 days
Archive Samples		16 per sample		Fish Tissue					-20°C	16x2ozWM	

[1] - The total number of samples in column 3 does not include QA samples such as PE samples and Matrix Spike/Matrix Spike Duplicate (MS/MSD) samples. The number of blind field duplicate (Dup) samples are included in the total number of samples in column 3. For example, of the 251 samples which will be measured for metals, 8 of the 251 samples will be blind field duplicate samples.
[2] - PE = Performance Evaluation Samples; MS/MSD = Matrix Spike/Matrix Spike Duplicate Sample; Dups = Blind Field Duplicate Samples.
[3] - Accuracy as measured in MS (matrix spike) and MSD (matrix spike duplicate) samples, which are measured at a frequency of 1:20 samples.
[4]- Precision as measured in MS (matrix spike) and MSD (matrix spike duplicate) samples, which are measured at a frequency of 1:20 samples.
[5] - Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991.
[6] - The number of filed samples will be revised by project members after January 6, 1997.

Table 2. Method 1613B PCDD/PCDF Target Compounds

Target Compound	CAS Number	Screening Tissue Concentration (STC) ng/Kg	Quantitation Limit[1] ng/Kg
2,3,7,8-TCDD	1746-01-6	0.002	0.2
1,2,3,7,8-PeCDD	40321-76-4	0.005	5
1,2,3,4,7,8-HxCDD	39227-28-6	0.024	5
1,2,3,6,7,8-HxCDD	57653-85-7	0.024	5
1,2,3,7,8,9-HxCDD	19408-74-3	0.024	5
1,2,3,4,6,7,8-HpCDD	35822-46-9	0.024	5
OCDD	3268-87-9	2.4	10
2,3,7,8-TCDF	51207-31-9	0.024	0.2
1,2,3,7,8-PeCDF	57177-41-6	0.048	5
2,3,4,7,8-PeCDF	57117-31-4	0.005	5
1,2,3,4,7,8-HxCDF	70648-26-9	0.005	5
1,2,3,6,7,8-HxCDF	57117-44-9	0.024	5
1,2,3,7,8,9-HxCDF	72918-21-9	0.024	5
2,3,4,6,7,8-HxCDF	60851-34-5	0.024	5
1,2,3,4,6,7,8-HpCDF	67562-39-4	0.24	5
1,2,3,4,7,8,9-HpCDF	55673-89-7	0.24	5
OCDF	39001-02-0	2.4	10

[1] - Quantitation limits listed for fish tissue samples are based on wet weight. A 50 gram fish tissue sample is used for extraction purposes.

Table 3. Method 1668 Toxic, Dioxin-Like, PCB Target Compounds

Target Compound[1]	Congener Number	CAS Number	Quantitation Limit[2] ng/Kg
3,3',4,4'-TCB	77	32598-13-3	2
2,3,3',4,4'-PeCB	105	32598-14-4	100
2,3,4,4',5-PeCB	114	74472-37-0	200
2,3',4,4',5-PeCB	118	31508-00-6	20
2',3,4,4',5-PeCB	123	65510-44-3	10
3,3',4,4',5-PeCB	126	57465-28-8	10
2,3,3',4,4',5-HxCB	156	38380-08-4	20
2,3,3',4,4',5'-HxCB	157	69782-90-7	20
2,3',4,4',5,5'-HxCB	167	52663-72-6	20
3,3',4,4',5,5'-HxCB	169	32774-16-6	20
2,2',3,3',4,4',5-HpCB	170	35065-30-6	20
2,2',3,4,4',5,5'-HpCB	180	35065-29-3	20
2,3,3',4,4',5,5'-HpCB	189	39635-31-9	20

[1] - Nomenclature for Polychlorinated Biphenyls:
TCB = Tetrachlorobiphenyl
PeCB = Pentachlorobiphenyl
HxCB = Hexachlorobiphenyl
HpCB = Heptachlorobiphenyl

[2] - Quantitation limits listed for fish tissue samples are based on wet weight. Quantitation Limits listed are estimated values due to high background levels of some selected standards and due to the lack of maturity of the method which was first proposed on October 4, 1995.

Table 4. Neutral Semivolatile Target Compound List

Target Compound	CAS Number	Risk Level [1] ug/Kg	Quantitation Limit ug/Kg [2]
1,2-Dichlorobenzene	95-50-1	1611.1	330
1,2,4-Trichlorobenzene	120-82-1	179	330
1,4-Dichlorobenzene	106-46-7	15.03	330
1,3-Dichlorobenzene	541-73-1	1593.2	330
2,2'-oxybis (1-Chloropropane) [3]	108-60-1	NC	330
2-Methylnaphthalene	91-57-6	NC	330
2-Chloronaphthalene	91-58-7	NC	330
2,4-Dinitrotoluene [3]	121-14-2	35.8	330
2,6-Dinitrotoluene [3]	606-20-2	0.53	330
4-Bromophenyl-phenylether	101-55-3	NC	330
4-Chlorophenyl-phenylether	7005-72-3	NC	330
Acenaphthene	83-32-9	1074.1	59
Acenaphthylene	208-96-8	NC	76
Anthracene	120-12-7	5370.4	22
Benzo(a)anthracene	56-55-3	0.34	10
Benzo(a)pyrene	50-32-8	0.049	10
Benzo(b)fluoranthene	205-99-2	0.4	10
Benzo(g,h,i)perylene	191-24-2	2.3	10
Benzo(k)fluoranthene	207-08-9	0.4	10
bis(2-Chloroethyl) ether	111-44-4	0.33	330
Chrysene	218-01-9	0.049	10
Dibenzo(a,h)anthracene	53-70-3	0.045	10
Dibenzofuran	132-64-9	NC	330
Fluoranthene	206-44-0	716	10
Fluorene	86-73-7	716	10
Hexachlorobutadiene [3]	87-68-3	4.6	330
Hexachloroethane [3]	67-72-1	17.9	330
Indeno(1,2,3-cd)pyrene	193-39-5	0.18	10
Naphthalene	91-20-3	716	59
Nitrobenzene	98-95-3	9	330
Phenanthrene	85-01-8	519.1	21
Pyrene	129-00-0	537	10

[1] - NC = Not Calculated due to lack of toxicity value for compound.
[2] - Quantitation limits listed for fish tissue samples are based on wet weight.
[3] - It is uncertain if this target compound will survive clean-up procedures.

Table 5. Chlorinated Phenolics Target Compound List

Target Compound [1]	CAS Number	Risk Level [2] ug/Kg	Quantitation Limit ug/Kg [3]
2-Chlorophenol	95-57-8	89.5	300
2,4,6-Trichlorophenol	88-06-2	32.6	300
2,4,5-Trichlorophenol	95-95-4	1790.1	300
2,4-Dichlorophenol	120-83-2	53.7	300
2,3,4,6-tetrachlorophenol	58-90-2		300
2,6-dichlorophenol	87-65-0		300
3,4-dichloroguaiacol	77102-94-4		300
3,4,6-trichloroguaiacol	60712-44-9		300
3,4,5-trichloroguaiacol	57057-83-7		300
4,6-dichloroguaiacol	16766-31-7		300
4,5,6-trichloroguaiacol	2668-24-8		300
4,5-dichloroguaiacol	2460-49-3		300
4-Chloro-3-methylphenol	59-50-7	NC	300
4-chloroguaiacol	16766-30-6		300
Pentachlorophenol	87-86-5	3	300
Tetrachloroguaiacol	2539-17-5		300

[1] -- Some compounds in this target compound list are expected to be lost during extract clean-up procedures.
[2] -- NC = Not Calculated due to lack of toxicity value for compound.
[3] -- Quantitation limits listed for fish tissue samples are based on wet weight.

Table 6. Inorganic Target Analyte List*

Target Compound	CAS Number	Risk Level [1] (mg/Kg)	Detection Limit (mg/Kg) [2]
Aluminum	7429-90-5	NC[2]	40
Antimony	7440-36-0	7.2	12
Arsenic	7440-38-2	0.21	2
Barium	7440-39-3	1253.1	40
Beryllium	7440-41-7	0.084	1
Cadmium	7440-43-9	9.0	1
Chromium	7440-47-3	89.5	2
Cobalt	7440-48-4	NC	10
Copper	7440-50-8	662.3	5
Lead	7439-92-1	7.7	0.6
Manganese	7439-96-5	89.5	3
Mercury	7439-97-6	5.4	0.1
Nickel	7440-02-0	358.0	8
Selenium	7782-49-2	89.5	1
Silver	7440-22-4	89.5	2
Thallium	7440-28-0	NC	2
Vanadium	7440-62-2	0.13	10
Zinc	7440-66-6	5370.4	4

[1] - NC = Not Calculated due to lack of toxicity value for compound.
[2] - Detection Limits listed for fish tissue samples are based on wet weight.

4.0 FIELD SAMPLING PROCEDURES

This section identifies the station locations (Section 4.1), target species and sample types (Section 4.2), sampling strategy (Section 4.3), field collection methods (Section 4.4), and handling of samples and documentation in the field (Section 4.5).

All of the field sampling for this project will be coordinated and conducted by EPA, Region 10, with assistance from the CRITFC tribes. The project leader and EPA FOM will thoroughly review the QAPP before sampling begins. Prior to sampling, the field team members will be familiar with:

- The responsibilities of each member of the field team
- Study objectives and time commitments for this project
- Collection permit requirements
- Site locations and collection equipment and gear needed at each site
- Proposed sampling dates and species of interest for each site location
- Composite sample size for each species and sample type
- Fish handling procedures and storage requirements.

4.1 STATION LOCATIONS

The CRITFC fish consumption survey (1) identified 102 fishing sites used by the four tribes in the Columbia River Basin. Due to resource constraints, all of these sites could not be sampled in Phase II of EPA's exposure study. The draft study design document referred to in Section 1.2 discusses in detail the process that was used to reduce the number of sites to be sampled to 13 sites. Initially, fishing sites that represented greater than 40 percent of each tribe's fishing use for resident and anadromous fish species were identified. This number of fishing sites (24 sites) was reduced to 8 sites by (1) selecting one site at the base of a watershed to represent the entire watershed for the Deschutes (site 98), Clearwater (site 96), and Umatilla (site 30) Rivers and (2) limiting the number of sites on the mainstem Columbia River to be sampled to sites 6, 7, 8, 9 and 18. Additional sites were added because they: are near local pollution sources of concern to the tribes (sites 48 and 49 on the Yakima River, and site 79 on the Salmon River); contain species of special concern to the tribe such as smelt (site 57 on the Cowlitz River); or provide needed geographical coverage (site 21 on the Willamette River). Use of this decision tree resulted in the selection of 13 sites for sampling.

Subsequent to the completion of the draft study design document, additional discussions were held with CRITFC tribal fisheries program managers and tribal staff. In these discussions, it was decided that for sites 9, 18, and 21, it would be easier to collect samples of salmon from nearby salmon hatcheries that supply salmon to the tribes. This is because recent data on fish runs suggested that low numbers of salmon may return to sites 9, 18, and 21. Also, using the fish returning to the hatcheries will help reduce some of the field collection time and sampling effort for this project. Therefore, at site 21, no salmon will be caught; they will instead be taken at site 21A (Dexter Hatchery on the McKenzie River). Salmon that were to be caught at site 9 will now be taken at site 14 (Priest Rapids Hatchery on the Columbia River); salmon that were to be caught at site 18 will now be taken at site 51 (Icicle Hatchery on the Wenatchee River). Other species will still be caught at sites 9, 18, and 21. An updated decision tree is shown in Figure 3 and now includes 16 sites . Site 14 will provide information on a local pollution source of concern, while sites 21A and 51 will provide the geographical coverage used in the decision tree. All of the fish species of interest and sampling locations are shown in Figure 4. The map of sampling locations from the June 17, 1996 revision of the QAPP is provided in Attachment 21.

The sampling locations in Figure 4 are not precise but rather indicate that area of the river system where fish will be collected. If an insufficient number of fish for a given species are collected from within the identified location, collection efforts may be extended to additional sections of the river as close as possible to the original location.

Whenever possible, the Global Positioning System (GPS) will be used to locate the sampling location (e.g. latitude and longitude) during fish collection efforts and this information will be transferred on to USGS topographical maps. If GPS positions cannot be obtained, then sampling locations will be determined using USGS topographical maps and the latitude and longitude recorded for this site . This information will be compiled in an appendix which will be included with the data report.

Figure 3. Decision Tree For Selection of Tissue Sampling Sites

Figure 4. Proposed Sampling Locations

4.2 TARGET SPECIES AND SAMPLE TYPE

Table 7 shows the locations, species, and sample types that will be measured during the entire 1996-1997 study. The selection of species to be collected was based primarily on consumption data presented in the CRITFC Fish Consumption Report. Input during the design conference in Portland and from the CRITFC tribal members was also considered. The primary target species selected are listed below:

Chinook salmon	**Oncorhynchus tshawytscha**
Coho salmon	**Oncorhynchus kisutch**
Steelhead trout	**Oncorhynchus mykiss**
Rainbow trout	**Oncorhynchus mykiss**
Mountain whitefish	**Prosopium williamsoni**
Lake whitefish	**Coregonus clupeaformis**
White sturgeon	**Acipenser transmontanus**
Walleye	**Stizostedion vitreum**
Largescale sucker	**Catostomus macrocheilus**
Bridgelip sucker	**Catostomus columbianus**
Eulachon (smelt)	**Thaleichthys pacificus**
Pacific lamprey	**Lampetera tridentata**

Table 9 shows the fish species that are consumed by tribal members and the fishing sites where fish are to be collected. Tissue samples for all consumed species except northern squawfish (Ptychocheilus oregonensis) and American shad (Alosa sapidissima) will be measured. These two species are consumed by only a small fraction (<2.7 percent) of adult tribal members.

If the primary species of aquatic organism can not be obtained, other species of fish will/may be substituted after consultation between the Project Manager, the Project Leader, the FOM, and CRITFC.

Four types of samples will be measured: whole-body (WB), fillet with skin (F_s), fillet without skin (F_w), and eggs (E). Whole-body samples were selected for several species to maximize the chances of measuring detectable levels of contaminants of concern and because data presented in the CRITFC fish consumption study show that tribal members may consume several fish parts in addition to the fillet (Table 10). Eggs from spring chinook, fall chinook, and steelhead will be measured because consumption data shows that salmonid eggs are widely consumed by tribal members (Table 10). Because of the high lipid levels in eggs, concentrations of hydrophobic organic chemicals may reach substantially higher levels than in other fish tissues. Salmonid heads were not designated as a matrix for compositing and analysis due to limited project resources and because the CRITFC fish consumption study did not indicate that most tribal members consumed large amounts of Salmonid heads on a frequent basis.

Table 7. Revised Sampling Design For the CRITFC Exposure Study [a]

Site No.	Location	Fish Species [b]	Res /Anad	Sample Type	Collection Period [c]	Collection Method	Repli-cates [d]	Number of Fish
57	Cowlitz River, lower	smelt	Anad	whole body	January 1997	dipnet	3	300
8	Columbia River, John Day Pool	steelhead	Anad	whole body	February 1997	gillnet	3	15
8	Columbia River, John Day Pool	steelhead	Anad	fillet with skin	February 1997	gillnet	3	15
8	Columbia River, John Day Pool	steelhead	Anad	eggs	February 1997	gillnet	3	0
56A	Klickitat River, lower	steelhead	Anad	whole body	February 1997	gillnet	3	15
56A	Klickitat River, lower	steelhead	Anad	fillet with skin	February 1997	gillnet	3	15
93	Snake River	steelhead	Anad	whole body	February 1997	gillnet	3	15
93	Snake River	steelhead	Anad	fillet with skin	February 1997	gillnet	3	15
6	Columbia River, Bonneville Pool	sturgeon	Res	fillet without skin	February 1997	deep water gillnet	3	3
7	Columbia River, Dalles Pool	sturgeon	Res	fillet without skin	February 1997	deep water gillnet	3	3
8	Columbia River, John Day Pool	sturgeon	Res	fillet without skin	February 1997	deep water gillnet	3	3
8	Columbia River, John Day Pool	sturgeon	Res	eggs	February 1997	deep water gillnet	3	0
96	Clearwater River, lower	sturgeon	Res	fillet without skin	March 1997	deep water gillnet	3	3
96	Clearwater River, lower	mountain whitefish	Res	whole body	March 1997	gillnet	3	45
96	Clearwater River, lower	mountain whitefish	Res	fillet with skin	35489	gillnet	3	45
98	Deschutes River	mountain whitefish	Res	whole body	35489	boat electrofish	3	45
98	Deschutes River	mountain whitefish	Res	fillet with skin	March 1997	boat electrofish	3	45
96	Clearwater River, lower	rainbow trout	Res	whole body	March 1997	boat electrofish, gillnet	3	45
96	Clearwater River, lower	rainbow trout	Res	fillet with skin	March 1997	boat electrofish, gillnet	3	45
98	Deschutes River	rainbow trout	Res	whole body	March 1997	boat electrofish	3	45
98	Deschutes River	rainbow trout	Res	fillet with skin	March 1997	boat electrofish	3	45
48	Yakima River, lower	steelhead	Anad	whole body	March 1997	dipnet (fish facility)	3	15
48	Yakima River, lower	steelhead	Anad	fillet with skin	March 1997	dipnet (fish facility)	3	15
96A	Clearwater River, lower	steelhead	Anad	whole body	March 1997	dipnet (hatchery)	3	15
96A	Clearwater River, lower	steelhead	Anad	fillet with skin	March 1997	dipnet (hatchery)	3	15
8	Columbia River, John Day Pool	sturgeon	Res	whole body	March 1997	deep water gillnet	3	3
98	Deschutes River	sucker	Res	whole body	March 1997	boat electrofish	3	30
98	Deschutes River	sucker	Res	fillet with skin	March 1997	boat electrofish	3	30
57A	Cowlitz River, upper	spring chinook	Anad	whole body	April 1997	dipnet (hatchery)	3	15
57A	Cowlitz River, upper	spring chinook	Anad	fillet with skin	April 1997	dipnet (hatchery)	3	15
18	Columbia River, at Rocky Reach	steelhead	Anad	whole body	April 1997	gillnet	3	15
18	Columbia River, at Rocky Reach	steelhead	Anad	fillet with skin	April 1997	gillnet	3	15
8	Columbia River, John Day Pool	lake whitefish	Res	whole body	May 1997	gillnet	3	45
8	Columbia River, John Day Pool	lake whitefish	Res	fillet with skin	May 1997	gillnet	3	45
8	Columbia River, John Day Pool	spring chinook	Anad	whole body	May 1997	gillnet	3	15
8	Columbia River, John Day Pool	spring chinook	Anad	fillet with skin	May 1997	gillnet	3	15
8	Columbia River, John Day Pool	spring chinook	Anad	eggs	May 1997	gillnet	3	0
21B	Willamette River, Middle Fork	spring chinook	Anad	whole body	May 1997	dipnet (hatchery)	3	15
21B	Willamette River, Middle Fork	spring chinook	Anad	fillet with skin	May 1997	dipnet (hatchery)	3	15
30	Umatilla River, lower	spring chinook	Anad	whole body	May 1997	gillnet	3	15
30	Umatilla River, lower	spring chinook	Anad	fillet with skin	May 1997	gillnet	3	15
56A	Klickitat River, lower	spring chinook	Anad	whole body	May 1997	gillnet	3	15
56A	Klickitat River, lower	spring chinook	Anad	fillet with skin	May 1997	gillnet	3	15
48	Yakima River, Prosser	spring chinook	Anad	whole body	June 1997	dipnet (fish facility)	3	15
48	Yakima River, Prosser	spring chinook	Anad	fillet with skin	June 1997	dipnet (fish facility)	3	15
9	Columbia River, Hanford	catfish	Res	whole body	July 1997	gillnet	3	30
9	Columbia River, Hanford	catfish	Res	fillet with skin	July 1997	gillnet	3	30
9	Columbia River, Hanford	lake whitefish	Res	whole body	July 1997	gillnet	3	45

Table 7. Revised Sampling Design For the CRITFC Exposure Study [a]

Site No.	Location	Fish Species [b]	Res /Anad	Sample Type	Collection Period [c]	Collection Method	Repli-cates [d]	Number of Fish
9	Columbia River, Hanford	lake whitefish	Res	fillet with skin	July 1997	gillnet	3	45
93	Snake River	rainbow trout	Res	whole body	July 1997	boat electrofish	3	45
93	Snake River	rainbow trout	Res	fillet with skin	July 1997	boat electrofish	3	45
51	Wenatchee River	spring chinook	Anad	whole body	July 1997	dipnet (hatchery)	3	15
51	Wenatchee River	spring chinook	Anad	fillet with skin	July 1997	dipnet (hatchery)	3	15
203	Palouse River	lake whitefish	Res	whole body	August 1997	gillnet	3	45
203	Palouse River	lake whitefish	Res	fillet with skin	August 1997	gillnet	3	45
24	Fifteen Mile Creek	lamprey	Anad	whole body	August 1997	dipnet	3	60
56	Klickitat River, upper	rainbow trout	Res	whole body	August 1997	backpack electrofish	3	45
56	Klickitat River, upper	rainbow trout	Res	fillet with skin	August 1997	backpack electrofish	3	45
79	South Fork Salmon River	rainbow trout	Res	whole body	August 1997	backpack electrofish	3	45
79	South Fork Salmon River	rainbow trout	Res	fillet with skin	August 1997	backpack electrofish	3	45
203	Palouse River	rainbow trout	Res	whole body	August 1997	boat electrofish	3	45
203	Palouse River	rainbow trout	Res	fillet with skin	August 1997	boat electrofish	3	45
9	Columbia River, above Snake	sturgeon	Res	fillet without skin	August 1997	deep water gillnet	3	3
203	Palouse River	sucker	Res	whole body	August 1997	boat electrofish	3	30
203	Palouse River	sucker	Res	fillet with skin	August 1997	boat electrofish	3	30
8	Columbia River, John Day Pool	walleye	Res	whole body	August 1997	gillnet	3	24
8	Columbia River, John Day Pool	walleye	Res	fillet with skin	August 1997	gillnet	3	24
8	Columbia River, John Day Pool	fall chinook	Anad	whole body	September 1997	gillnet	3	15
8	Columbia River, John Day Pool	fall chinook	Anad	fillet with skin	September 1997	gillnet	3	15
8	Columbia River, John Day Pool	fall chinook	Anad	eggs	September 1997	gillnet	3	0
56A	Klickitat River, lower	fall chinook	Anad	whole body	September 1997	gillnet	3	15
56A	Klickitat River, lower	fall chinook	Anad	fillet with skin	September 1997	gillnet	3	15
14	Columbia River, near Priest Rapids	fall chinook	Anad	whole body	October 1997	dipnet (hatchery)	3	15
14	Columbia River, near Priest Rapids	fall chinook	Anad	fillet with skin	October 1997	dipnet (hatchery)	3	15
30A	Umatilla River, lower	fall chinook	Anad	whole body	October 1997	dipnet (holding pond)	3	15
30A	Umatilla River, lower	fall chinook	Anad	fillet with skin	October 1997	dipnet (holding pond)	3	15
48	Yakima River, Prosser	fall chinook	Anad	whole body	October 1997	dipnet (fish facility)	3	15
48	Yakima River, Prosser	fall chinook	Anad	fillet with skin	October 1997	dipnet (fish facility)	3	15
8A	Columbia River, at Umatilla River	walleye	Res	whole body	July 10, 1996	boat electrofish, gillnet	1	8
8A	Columbia River, at Umatilla River	walleye	Res	fillet with skin	July 10, 1996	boat electrofish, gillnet	3	24
101	Umatilla River, upper	mountain whitefish	Res	whole body	July 11, 1996	backpack electrofish	3	27
101	Umatilla River, upper	mountain whitefish	Res	fillet with skin	July 11, 1996	backpack electrofish	3	27
101	Umatilla River, upper	rainbow trout	Res	whole body	July 11, 1996	backpack electrofish	2	40
8A	Columbia River, at Umatilla River	sucker	Res	whole body	July 11, 1996	boat electrofish, gillnet	3	36
8A	Columbia River, at Umatilla River	sucker	Res	fillet with skin	July 11, 1996	boat electrofish, gillnet	1	4
101	Umatilla River, upper	rainbow trout	Res	whole body	July 13, 1996	backpack electrofish	2	60
48	Yakima River, lower	sucker, bridge lip	Res	whole body	July 15, 1996	dipnet (fish facility)	3	21
48	Yakima River, lower	sucker, large scale	Res	whole body	July 15, 1996	dipnet (fish facility)	3	21
21	Willamette River, lower	lamprey	Anad	whole body	June 20, 1996	dipnet	3	60
49	Yakima River, upper	rainbow trout	Res	fillet with skin	September 11, 1996	boat electrofish	3	21
49	Yakima River, upper	rainbow trout	Res	whole body	September 12, 1996	boat electrofish	3	21
49	Yakima River, upper	sucker	Res	whole body	September 12, 1996	boat electrofish	3	15

Table 7. Revised Sampling Design For the CRITFC Exposure Study [a]

Site No.	Location	Fish Species [b]	Res /Anad	Sample Type	Collection Period [c]	Collection Method	Repli-cates [d]	Number of Fish
49	Yakima River, upper	sucker	Res	fillet with skin	September 12, 1996	boat electrofish	3	15
8A	Columbia River, at Umatilla River	sucker	Res	fillet with skin	September 9, 1996	boat electrofish	3	24
						Sample Total	276	2560

[a] -Table 7 has been modified to reflect what EPA field crews will attempt to complete without additional resources, equipment, and ESA collection permit from the CRITFC organization. This field sampling effort will require the EPA field crew to acquire state collection permits to complete these objectives.

[b] - Samples from all species are composites (composites samples consist of 20 lamprey each and 8 each for other fish species).

[c] - Dates reflect suggested sampling periods.

[d] - Number of samples assumes each tissue sample is performed in triplicate.

Table 9. Percentage of Adult Tribal Members Consuming Proposed Target Species and Species Collection Sites

Species	Weighted Percent That Consume the Species	Proposed Fishing Sites	
		Site Numbers	Site Locations (Rivers)
Salmon	92.4%	8, 9, 14*, 21A*, 30, 51*	Columbia, McKenzie, Umatilla, Wenatchee,
Lamprey	54.2%	21, 6	Willamette, Columbia
Trout [a]	70.2%	98, 8, 18, 30, 48, 49, 96*, 79	Deschutes, Columbia, Umatilla, Yakima, Clearwater, Salmon
Smelt	52.1%	57	Cowlitz
Whitefish	22.8%	8, 30, 96	Columbia, Umatilla, Clearwater
Sturgeon	24.8%	6, 7, 8, 9, 96	Columbia, Clearwater
Walleye	9.3%	8	Columbia
Sucker	7.7%	98	Deschutes
Squawfish	2.7%	none	none
Shad	2.6%	none	none

Source: Modified from CRITFC (1).

[a] Rainbow Trout and Steelhead.
* Hatchery Site.

Table 10. Columbia River Inter-Tribal Fish Commission Exposure Study: Adult Consumption of Fish Parts

Species	Parts											
	Fillet		Skin		Head		Eggs		Bones		Organs	
	N	Weighted % That Consume	N	Weighted % That Consume	N	Weighted % That Consume	N	Weighted % That Consume	N	Weighted % That Consume	N	Weighted % That Consume
Salmon	473	95.1%	473	55.8%	473	42.7%	473	42.8%	473	12.1%	470	3.7%
Lamprey	249	86.4%	251	89.3%	250	18.1%	250	4.6%	250	5.2%	250	3.2%
Trout	365	89.4%	365	68.5%	365	13.7%	364	8.7%	365	7.1%	362	2.3%
Smelt	209	78.8%	209	88.9%	210	37.4%	209	46.4%	210	28.4%	206	27.9%
Whitefish	125	93.8%	124	53.8%	125	15.4%	125	20.6%	125	6.0%	124	0.0%
Sturgeon	121	94.6%	121	18.2%	121	6.2%	121	11.9%	121	2.6%	121	0.3%
Walleye	46	100%	46	20.7%	46	6.2%	46	9.8%	46	2.4%	46	0.9%
Sucker	15	89.7%	15	34.1%	15	8.1%	15	11.1%	15	5.9%	15	0.0%
Squawfish	42	89.3%	42	50.0%	42	19.4%	42	30.4%	42	9.8%	42	2.1%
Shad	16	93.5%	16	15.7%	16	0.0%	16	0.0%	16	3.3%	15	0.0%

Source: CRITFC (1).

Contaminant levels in various fish parts (i.e., whole-body, fillet, and eggs) will be estimated so that this information can be used to provide guidance on how to prepare fish, or what parts should be avoided, in the event that contaminant levels exceed levels that warrant concern. In addition, the conversion factors developed from these data (e.g., whole-body-fillet and whole-body-egg ratios) may assist in the comparison of the data from this study with other historical data that exist from the Columbia River Basin. Table 7 indicates that most of the comparisons of contaminant levels in different fish body parts will occur at Site 8 in the Columbia River between the McNary and John Day dams. This site was selected because of its importance as a fishing site for all four CRITFC member tribes.

4.3 SAMPLING STRATEGY

The sampling strategy proposed for this study design is consistent with guidance provided in the document entitled: **Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume I: Fish Sampling and Analysis** (4). For all fish species except white sturgeon, three replicate composite samples will be measured from each collection site. For white sturgeon, composite samples will not be taken. Instead three individual fish will be measured from each collection site. The planned number of fish per composite will likely vary for different species: 100 individuals per composite for smelt, 20 individuals per composite for lamprey, 8 individuals per composite for resident (non-salmonid) species, and 5 individuals per composite for salmon and steelhead. U.S. EPA (4) recommends that 3 to 10 individuals should be collected for a composite sample for each target species and that the same number of individual organisms should be used to prepare all replicate composite samples for analysis of contaminants for a given target species at a given site. Several ongoing fish contaminant studies in the Columbia River Basin are compositing 8 individuals per sample, so the use of this number would simplify comparisons with other available data. Because of the small size of lamprey and smelt, a composite of 8 individuals would not provide enough tissue for all chemical analyses; therefore a nominal value of 20 individuals per composite was suggested by the Design Conference attendees for smelt and lamprey, respectively. To ensure adequate sample volume for analyses, EPA, Region 10, decided to increase the composite size for smelt to 100 fish. Design Conference attendees felt that the number of individuals per composite for salmon and steelhead should be reduced from 8 to 5 (some individuals suggested 3) because of concerns about the ability to collect sufficient numbers of fish, and because it was felt that the study should strive to minimize impacts on these fish stocks.

At the Scoping Meeting, it was recommended that if possible, all fish used in a composite be female. This recommendation was made because eggs are to be collected for some of the sampling locations and because it was thought that females have a higher lipid content (and, therefore, potentially a higher contaminant level for lipid soluble contaminants). However, recent data collected by the Lower Columbia Bistate Program suggest that, for chinook, coho, and steelhead, males have the higher lipid content. The Bistate Program measured the lipid content and contaminant levels for male and female fish for these three species. For all three species, percentages of lipids were substantially higher in male fillet as is specified in the following table.

SPECIES	PERCENT LIPIDS (MALE)	PERCENT LIPIDS (FEMALE)
Chinook	3.51%	0.72%
Coho	1.67%	0.85%
Steelhead	4.06% 4.82%	2.87%

Based upon this lipid data and upon the fact that the Native Americans eat what they collect (i.e. both males and females), the decision was made to collect random samples of fish (by sex) for each composite rather than all females. The exception to this will be at site #8. At this site, for fall and spring chinook and steelhead, the fish to be used for the fillet with skin composites will be as follows:

Composite 1 - 5 fish, all male
Composite 2 - 5 fish, all female
Composite 3 - random

This will provide information on the lipid content of males and females of these three species. Eggs that are to be collected from these species at this location will be taken from Composite Number Two, above.

Collection periods for each species have been tentatively assigned and are given in Table 7. According to U.S. EPA guidance (4), the collection period should ideally avoid the spawning period of the target species because many fish are subject to stress during spawning. However, because eggs will be collected from salmonid species and because the CRITFC tribes fish for salmonids when they are spawning, the typical spawning period for these species will be targeted. For resident species, collection periods have been proposed so that spawning periods can be avoided. For white sturgeon, the proposed collection period is consistent with seasons established in previous years.

For each target species composite, a single size class will be targeted at the site. Because the concentrations in fish for some pollutants (e.g., PCBs and mercury) have been shown to increase with age and size, an attempt will be made to collect a composite that represents the larger fish being caught at the sampling site during the sampling period. Therefore, the selection of fish for the composite will, when possible, adhere to the following two criteria:

(1) Composites will be comprised of fish that are in the upper 75% of fish length of those fish being caught by the CRITFC tribes near the sampling location, and;

(2) Composites will comply with EPA guidance (reference 4) which recommends that the smallest individual in a composite be no less than 75% of the total length of the largest individual.

Replicate composite samples for a target species should be as similar to each other as possible. Therefore, if possible, the relative difference between the average length of individuals within any composite sample from a given site as well as the average of the average lengths of individuals in all composite samples from that site will not exceed 10 percent.

This goal may not be possible for composite samples if (1) fish populations are low and (2) endangered species considerations for salmon limit the number of fish that should be caught. In those cases where the above goal is unattainable during the time scheduled for sampling, composites will be prepared using available fish. These composites will represent all sizes of fish captured at the different sampling sites. In all cases, the total length and weight of each fish in the composite sample will be recorded.

4.4 FIELD COLLECTION METHODS

Sampling methods for finfish include: electrofishing, hand collection, hatchery collection, trapping at dams, dip netting, and gill netting. The preferred method will be dependent on the conditions at the sampling site, selected species, and legal constraints. Collection of fish by any techniques will be controlled by the stipulations of the federal, state and tribal permits. Copies of permits should be in the possession of the field sampler at all times.

The EPA FOM and his EPA alternate are qualified boat operators as defined in EPA Region 10's "Boat Operating Policy" (Attachment 2). Both will ensure that the necessary safety equipment is available for all sampling team members on all EPA boats used and that emergency information is available (e.g., local hospitals and police). Sampling team members will also be briefed on boat safety prior to launching any EPA vessel. The safety procedures that will be followed for electrofishing are provided in Attachment 3. At some locations, boats and equipment owned by the CRITFC tribes may be used for sampling.

4.4.1 Electrofishing

Electrofishing is considered to be the most efficient method for collecting a variety of species in large rivers because it is easily standardized and less selective than alternative gear. However, electrofishing is generally not effective in capturing fish that are at depths greater than about 10 feet, therefore alternative methods, such as gillnetting, will need to be used for some species. In this project the boat mounted electro shocker will be used in the deeper rivers. Some of the smaller rivers selected (i.e., Deschutes, Umatilla, South Fork of the Salmon) may not be deep enough to use the boat for electrofishing. In these smaller rivers, sampling will be done using electro backpack shocking equipment allowing for the selection of the fish species of interest. It is anticipated that steelhead, rainbow trout, whitefish, and sucker from selected locations (as shown in Table 7) will be captured by electrofishing. Only fish that appear to be in the desired target size range (see below) will be brought aboard using a dip net. The fish that are not netted will be allowed to recover from the electroshocking pulse by shutting off electrofishing equipment until fish swim away from the boat.

4.4.2 Gillnetting

Gill nets capture fish by entanglement. They are particularly well-suited for the capture of highly mobile fish (e.g., salmonids) which are not easily captured by electrofishing. For this project, sinking gill nets (approximately 100 ft long by 6 ft or 12 ft deep) will be used, each of which consists of variable mesh (2 to 6 inch diameter) monofilament line attached to cork and lead lines. The nets will be anchored with lead mushroom weights and marked with the appropriate information identifying who the nets belong to and how they are being used (i.e., research). Flashing lights should be attached to either end to help mark net deployment areas.

Gillnets will be deployed and monitored during the fishing efforts for both day and night operations. After several hours the nets will be retrieved and the captured fish collected. All non-target fish species and all targeted fish species that are not within the desired size category will be returned to the water, whether dead or alive. A record will be kept of the catch of each gillnet set. It is anticipated that chinook, sturgeon, steelhead, whitefish, walleye, and rainbow trout from selected sampling sites will be captured by gillnetting (see Table 7).

4.4.3 Trap/Dam

At the barrier dam on the Umatilla River, fish have no access through the dam and are trapped behind weirs. Samples of selected fish species (e.g., steelhead and salmon) may be taken from these weirs using dipnets.

4.4.4 Dipnet

Dipnets may be used in areas where the migrating fish, such as smelt, steelhead, salmon and lamprey, are following the shoreline of the river. Dipnets are usually made with small cotton mesh (e.g. ½" to 3") and used to dip up fish in small confined areas such as shallow pools or water falls. The sampling nets will need to be monitored at all times to be most effective. Once a fish is caught, the dip net will be pulled to the surface and the fish removed. Only the fish selected for the project will be retained and other species will be released.

4.4.5 Hatchery

Specific fish returning to the hatchery can be targeted for collection and retrieved from the holding pond. This sampling effort will be coordinated with the hatchery management personnel so that the fish can be taken from the holding pond area before their eggs and sperm are removed and before any type of chemical treatment has been applied.

4.4.6 Hand Collection

The hand collection method of sampling will be used in and around the Willamette Falls for lamprey. As the lamprey migrate over the falls area, they can be collected off the rocks and from shallow pools with small nets or by hand.

4.5 FISH SAMPLE HANDLING IN THE FIELD

4.5.1 Sample Integrity

The EPA FOM or his EPA alternate will be present at all times when fish are collected in order to assure sample integrity.

Sample integrity requires that fish be handled in a manner that prevents loss of contaminants already in the fish and prevents extraneous tissue contamination. Loss of contaminants already in the fish tissue will be prevented in the field by ensuring that the skin on fish specimens has not been lacerated by the sampling gear. Sources of extraneous tissue contamination include contamination from dirty hands, sampling gear, greasy cables, spilled engine fuel, engine exhaust, dust, ice chests and ice used for cooling.

The FOM will identify all potential sources of contamination in the field and take appropriate steps to minimize or eliminate them. The FOM will observe the following practices (and others as indicated by professional judgement) as well as provide training to tribal members who are assisting in fish collection in these practices: (1) Caught fish will only be placed on clean surfaces, such as aluminum foil. (2) Ice chests will be cleaned prior to any sampling activities. (3) Samples will be placed in waterproof plastic bags to avoid contamination from melting ice. (4) Sampling equipment, such as gillnets and dipnets, will be free from contaminants such as oils, grease and fuels. (5) All utensils or equipment used directly in handling fish (e.g., such as fish hooks, measuring boards and fish clubs) will be cleaned in the laboratory prior to each field sampling effort and placed in aluminum foil. (6) The field collection team will clean this equipment between sampling sites by rinsing with ambient water and rewrapping in aluminum foil.

4.5.2 Handling Of Field Samples During Collection

Upon retrieval from the sampling equipment, each fish will be identified by species by personnel familiar with the taxonomy of
the fish in the Columbia River Basin. The FOM will assure that a taxonomic key is readily available at all times. Once a target species is caught, the length of the fish will be measured to ensure that it meets the target size class as defined in Section 4.3. Based upon size of fish caught in the field, the acceptable size range of fish will be determined by the FOM and documented using a Sample Alteration Form (see Attachment 17). Those fish that do not meet the target size class will be released unharmed. The fish that do meet the target size class will be subdued by a sharp blow or blows to the base of skull. All individual fish (with the exception of lamprey and smelt) that are kept will be assigned a unique identification number (EPA Sample #) consisting of an numeric eight digit code XXXXXXXX. The fish will then be assigned to one of the three composite samples for that location which will also have a unique identification number. These numbers will be chosen to be consistent with EPA Region 10's sample management tracking system. Selected specimens will be photographed. For lamprey and smelt, each fish will be placed into one of three composite groups (approximately 20 per composite for lamprey and 100 per composite for smelt) and each composite group assigned an identification number.

The FOM will wrap each whole fish (with the exception of lamprey and smelt) in clean heavy-duty aluminum foil. The whole fish will then be placed into a plastic bag and the bag will be tied.

For lamprey and smelt, the composite group will be wrapped in aluminum foil and tied in a plastic bag. The FOM will immediately pack the bagged fish sample on ice (preferably dry ice) in clean ice chests to start cooling the fish down.

4.5.3 Documentation During Fish Collection

The FOM will be personally responsible for the care and custody of the fish samples until they are properly transferred or dispatched to the storage and/or filleting facility or to the subcontract laboratory. He will also determine whether custody procedures are followed properly during the field work and will decide if additional samples are required.

Documentation for fish collection consists of information that must be provided: (1) on the Field Record Form; (2) in the Sampler's Notebook; (3) on the Sample Identification/Chain of Custody Tag, and (4) on the Chain-of-Custody Form.

Field Record Form - EPA has developed a standard Field Record Form (attachment 4) that will be filled out by EPA at each sampling location. The information listed below will be included on this Field Record Form:

Geographic location (latitude and longitude) using Global Positioning System
Species name
Date and time
Method of collection (e.g., gill net, trap, electrofish, etc.)
Station number
Sample identification number / numbers
Composite sample number
Weather conditions (e.g., cloud cover, rain or shine, windy)
Water depth of capture (feet)
Sex of species
Evidence of hatchery markings (e.g.., fin clips, tags)(under "Comments")
Total fish length (in metric units)
Total fish weight (in metric units to the nearest gram)
Sampling crew names
Type of vessel
External marks or gross physiological abnormalities noted (under "Comments")

Sampler's Notebook - The sampler's notebook will include the same information that is on the Field Record Form. In addition, the Sampler's Notebook will be used to document any unusual activities or problems encountered in the field that would be useful for the Project Leader and Manager to be aware of when data quality is being evaluated. It will also include a record of any photographs taken in the field.

Sample Identification/Chain of Custody Tag - A waterproof Sample Identification/Chain of Custody Tag (SI/COC Tag) (Attachment 5) should be completed in indelible ink for each individual fish (or composite for lamprey and smelt) and taped to each aluminum-foil-wrapped

specimen(s) before placing the specimen(s) in a plastic bag in the field. This tag will include the following information: the project name/code, station location/number, sampling date and time, species name, fish sample and/or composite number, sample length and weight, and the name, phone number, and signature of the sampler.

If a fish sample tag is lost during shipment or a tag is never created, the FOM will write a statement detailing how the sample was collected, stored, and transferred to the laboratory. The statement will include all pertinent information, such as entries in field logbooks regarding the sample, whether the sample was in the sample collector's physical possession or in a locked compartment until hand transported to the laboratory, etc.

Chain-of Custody Form - A Region 10 Chain-of-Custody Form (COC Form) (Attachment 6) will be completed in indelible ink for each shipment that is made. These COC forms will be enclosed in plastic and taped to the inside lid of the cooler. The information on this form will be used to track all samples from field collection to receipt at the subcontract laboratory.

5.0 SAMPLE STORAGE

5.1 STORAGE PROCEDURES

Once fish are caught, the FOM will immediately pack the bagged fish samples in ice (preferably dry ice) to start cooling the fish down. If fish are to be filleted the same day they are caught, they will not be frozen. Fish that will not be filleted the day they are caught or whole fish samples that are not shipped to the subcontract laboratory the day they are caught will be transported to the EPA Laboratory or to another prearranged locations (e.g. local fish hatcheries) having freezer space available. This freezer must have a temperature less than or equal to $\leq$-20 ^{o}C and must be secured. Fish will be completely frozen before any shipping occurs.

5.2 DOCUMENTATION

The COC Forms and SI/COC Tags described in section 4.5.3 will remain with the stored samples until samples are removed for filleting and/or shipping. In addition, the FOM will include information in the Sampler's Notebook on:

- Sample storage location and contact person
- Compliance of storage location with EPA Chain of Custody procedures if storage location is not the Region 10 EPA laboratory
- Freezer temperature

6.0 FILLETING OF FISH

The FOM or EPA Region 10 staff (with oversight by the FOM) will fillet selected fish samples. The samples to be filleted are identified in Table 7. Filleting will be done at the EPA Laboratory or at a field laboratory that allows for the appropriate quality control procedures to be followed (e.g., a fish hatchery or EPA's mobile trailer.)

6.1 FILLETING PROCEDURES

Fish will be handled following the guidance provided in sections 7.2.1 (General Considerations) and 7.2.1.3 (Samples for Both Organics and Metals Analyses) in Reference 4 (see Attachment 7 for a copy of these sections of Reference 4). Fish will be partially thawed prior to filleting. If rupture of organs is noted for an individual fish, the specimen will be eliminated from the composite sample. For scaling and filleting, the methods described in sections 7.2.2.6 and 7.2.2.7 and illustrated in Figure 7-3 of Reference 4 will be followed (see Attachment 8 for a copy of these sections and the figure in Reference 4). Labeling of fish filleted for compositing will be done as described in the next paragraph.

The FOM will create composites of fillets (with and without skin) using the fillet from the right side of each fish (the "F1s")(right side to be determined from the perspective of the direction in which the fish would swim). This composite will be wrapped in clean aluminum foil and placed in a plastic bag. The FOM will wrap the left side fillet from each fish separately in heavy duty aluminum foil and add the two digit identifier "F2" to the end of the sample number for this fillet. The individual fillets (the "F2"s) that will not be ground and composited will be placed in individual plastic bags with the composite identification number, the individual identification numbers, and the date of resection. The FOM will arrange for shipment of the F2s to the EPA Region 10 laboratory for storage.

6.2 DOCUMENTATION PROCEDURES DURING FILLETING

Documentation for fish filleting consists of information that must be provided: (1) on a Sample Processing Record; (2) in the Filleter's Notebook; and (3) on the Sample Identification/Chain of Custody Tag.

6.2.1 Sample Processing Record

Sample processing records will be kept for each individual sample (sturgeon) or composite of whole fish, fillets and eggs. The record (Attachment 9) will include the following information:

- Information on sample type and species name
- Unique sample number for individual fish and/or fish (egg) composite number (identical to that number assigned during sampling)
- Weights of unprocessed individual fillets and egg skeins

(Additional information to be added after sample homogenation is included on these forms - this will be completed by the subcontract laboratory)

6.2.2 Filleter's Notebook

The filleter (the FOM or Region 10 designee) will record the information described above for the Sample Processing Record in a Filleter's Notebook. Additional information to be recorded in the Filleter's Notebook is as follows:

- Evidence of hatchery markings on fish (e.g., fin clips) in addition to those noted in the field
- Incidence of external abnormalities (e.g., fin erosion, skin ulcers, skeletal anomalies, tumors) in addition to those noted during field sampling
- Incidence of internal abnormalities if any
- Record of scales and/or pectoral fins collected.

Scales for age determination will be collected for all fish, except for smelt, lamprey, and sturgeon. Lamprey do not have scales and smelt are too small to obtain scales. For sturgeon, one pectoral fin will be removed by EPA prior to filleting and this fin will be shipped to Oregon Department of Fish and Wildlife for ageing. Otoliths may be also taken for selected fish to verify aging done using scales. Scales (and otoliths if collected) will be placed in small jars and preserved with ethanol. Pectoral fins will placed in plastic bags and frozen until ageing. Each scale or fin sample taken will be given a matching EPA sample number.

6.2.3 Sample Identification/Chain-of-Custody Tag

After filleting, SI/COC Tags (containing the information described in section 4.5.3) will be attached to the aluminum foil on individual fish fillet (sturgeon and the individual "F2s" from fish forming a composite) and on the combined composite fillets (the combined "F1s"). These will then be placed in plastic bags.

7.0 SHIPMENT OF SAMPLES AND RECEIPT BY SUBCONTRACT LABORATORY

In preparation for shipping, the FOM will pack whole fish, fillets, and egg samples securely inside a cooler with dry ice. The cooler will be closed, fiber tape will be wrapped completely around it, and a custody seal (shown in Attachment 10) will be attached so that it must be broken when the cooler is opened. All fish samples will be packaged and shipped to the subcontract laboratory (for further processing) or to the Region 10 EPA Laboratory (for storage of the "F2" fillets and of pectoral fins and scales) via overnight delivery using Federal Express.

As identified in "Dangerous Goods Regulations" (36th Edition, January, 1995, International Air Transport Association), the FOM will assure that appropriate dry ice labels (shown in Attachment 10) will be affixed to each shipping container. These same procedures must be followed by the subcontract laboratory when sending processed samples to the EPA laboratory for analysis and archiving (see Section 8.6).

The EPA FOM will notify the Tetra Tech contact person when samples will be shipped. The contact person will be given sample ID numbers, number of ice chests being sent and species of fish being sent in each mailing. Upon arrival at the laboratory, fish tissue may be distributed immediately to a technician for processing. If they are not processed immediately, they must be stored in a freezer at $\leq$-20^{o} C until they are removed for processing.

7.1 DOCUMENTATION REQUIREMENTS

The original Region 10 Chain-of-Custody Forms will be signed by the FOM and enclosed in plastic and taped to the inside lid of one cooler of each group of coolers shipped at one time. A custody seal will be attached to each cooler so that it must be broken when the cooler is opened. The Sample Processing Records and the SI/COC Tags for each sample will be shipped at the same time. In addition, one photocopy of all of the paperwork sent to the subcontract laboratory will be sent to the Tetra Tech contact person via Federal Express or FAX and one copy will be retained by the FOM.

Upon receipt by the subcontract laboratory, the return delivery receipts and chain-of-custody procedures listed in Attachment 11 should be followed. The return delivery receipt will be sent to Tetra Tech. A copy of the Chain-of-Custody Form for each shipment will be delivered by the subcontract laboratory to EPA (the WAM) within 7 calendar days of receipt of each shipment of samples.

In addition to the written record required by Attachment 11, the subcontract laboratory will contact the FOM after they have received the samples to let the FOM know if sample integrity was maintained during shipment. The following information will be communicated to the FOM by telephone or FAX within 24 hours after samples are received: (1) condition of the samples upon arrival at the laboratory (e.g. to ensure sample degradation has not occurred during

shipment); (2) time delays (e.g., not arriving the next day); (3) condition of chain-of-custody seals. A project file including a copy of all Chain of Custody forms, field notebooks, *etc.*, will be maintained by the Project Manager at the EPA Region 10 Seattle office.

8.0 HOMOGENIZATION OF INDIVIDUAL FISH AND COMPOSITES AND DISTRIBUTION OF HOMOGENIZED SAMPLES

Upon receipt of fish and egg samples from the FOM, the subcontract laboratory will homogenize the samples, prepare sample aliquots, and distribute these aliquots to the appropriate analytical laboratories for analyses.

8.1 GENERAL CONSIDERATIONS FOR HANDLING SAMPLES

Fish samples and homogenized samples will be handled following the guidance provided in sections 7.2.1 (General Considerations) and 7.2.1.3 (Samples for Both Organics and Metals Analyses) of Reference 4 (see Attachment 7 of this QAPP for a copy of these sections of Reference 4). An additional requirement is that the Hobart grinder specified below must be completely taken apart and the auger, auger housing, orifice plate, and any implement used to push tissue through the grinder be cleaned after each sample (individual fish or fish/egg composite) has been homogenized.

8.2 GENERAL CONSIDERATIONS FOR PREPARING COMPOSITES

Composite samples may be prepared using two different methods. In the first method (the "individual" method), each individual fish or fish fillet that is to be part of a composite is homogenized separately. Equal weights of each individual fish homogenate are then compiled into a composite and homogenized again. The individual method is designed to provide information on the mean concentration of contamination in fish tissue for the fish population that is being sampled. In the second method (the "batch method"), all of the fillets or whole fish that are to be part of a composite are homogenized together. The batch method provides information on the weighted mean of the concentration in the batch sampled.

For this project, composites will be homogenized by the subcontract laboratory using the batch method. Information on the fish consumption habits of tribal members suggest that once fish are caught, the entire fish is consumed. Therefore, the information on contaminant levels provided by the batch method (which includes information from the entire fillet of each fish in a composite) will provide a more appropriate estimate of exposure for the Native Americans. It is expected that since every attempt will be made to ensure that fish that make up a composite sample will be similar in size (i.e., the smallest individual will be no less than 75% of the total length of the largest individual), the mean concentrations generated by the batch method will likely be similar to that generated using the individual method.

The batch method is also easier to implement in the laboratory because it saves sample preparation time and resources and maximizes the amount of tissue available after grinding

smaller fish. This is because tissue from smaller fish often remain inside the grinder due to the small volume of sample going through the grinder.

8.3 SAMPLE HOMOGENIZATION

Whole fish, fish fillets, and eggs should be ground and homogenized using a Hobart Model 84186 commercial meat grinder. If possible, the blades of the Hobart should be made of titanium or tantalum rather than stainless steel since stainless steel blades have been found to be a potential source of nickel and chromium contamination (due to abrasion at high speeds) and should be avoided. While an orifice size of 3/16th inch is recommended, orifice sizes up to 1/4th inch could be used.

Grinding of tissue is easier when it is partially frozen. Chilling the grinder briefly with a few chips of dry ice may also keep the tissue from sticking to it.

For larger fish samples, the fish tissue should first be cut into small pieces no larger than 2.5 cm cubes and then the fish tissue cubes from all of the fish that make up one composite sample should be combined and ground in the equipment specified. After the first grinding, the ground material should be divided into quarters, opposite quarters mixed together by hand, and the two halves mixed together. **At a minimum, each composite sample should be run through the grinder and hand mixed three times**. If chunks of tissue are present at this point, the grinding and homogenization should be repeated until the composite sample appears to be homogenous. No chunks of tissue or pieces of skin should remain because these may not be extracted or digested efficiently.

Egg samples sent to the subcontract laboratory by EPA should be ground using the procedures given in the paragraphs above for fish samples **(i.e., ground a minimum of three times)**.

The subcontract laboratory will prepare an adequate amount of homogenate to meet the requirements for analysis as specified
in Table 1 (column titles "Containers").

8.4 SAMPLE DISTRIBUTION

The subcontract laboratory must prepare sample aliquots (as described in section 8.5) immediately after homogenization is completed and then distribute these sample aliquots to the appropriate laboratory for chemical analyses. Unless aliquots are to be measured immediately, they must be frozen and stored in a secure location at $\leq$-20^{o} C until transfer to the EPA laboratory for analysis or until analyses are begun by the subcontract laboratory. If adequate homogenate is available, approximately 400 grams of the unused portion of each homogenate (i.e., that not put into sample aliquot jars) should be placed into each of two (2) wide mouth glass 16 ounce jars and stored at $\leq$-20^{o} C by the subcontract laboratory for 30 calendar days. This will ensure that adequate sample homogenate is available for EPA analysis and archiving in case the aliquots sent to the EPA

laboratory are lost or damaged during shipment. The shipping directions found in Section 7.0 (i.e., overnight shipping on dry ice) should be followed. Glass jars should be securely packed to avoid breakage during shipment.

8.5 SAMPLE CONTAINERS AND LABELS

The laboratory will place approximately fifty (50) grams of homogenized sample into each of 26 properly cleaned wide mouth
2 ounce glass sample jars. The laboratory must leave sufficient headspace in each jar such that expansion during freezing does not cause the jar to break. As shown on Table 1, a total of 4 jars (2 for PCDDs/PCDFs analyses and 2 for dioxin-like PCB analyses) will be retained by the subcontract laboratory for analyses of PCDDs/PCDFs and toxic, dioxin-like, PCBs. The laboratory will send the 22 remaining jars to Region 10 EPA's laboratory.

EPA will use twelve (12) of these jars for the analyses of pesticides/PCBs, semivolatiles, PAHs, Target Analyte List (TAL) inorganics, mercury and arsenic. The sample jar distribution for measurements by the EPA Manchester Laboratory will be as follows: 2 for pest/PCB (100 grams), 2 for PAHs and semi-volatiles (100 grams), 2 for TAL inorganics, arsenic, and mercury (100 grams), and 16 for archive (800 grams).

If resources become available, some of the archived material will be used for analysis of selected radionuclides.

The laboratory must affix bottle labels firmly to each sample container and lid. The laboratory must keep these bottles and lids dry and empty before labeling so that the gummed label can be securely attached to the side of the container and the tape stuck to the lid. Each container label and lid tape should be filled out with the appropriate sampling information. The following list identifies the information that must be written on each container and lid label:

LID LABEL
* EPA Composite Sample Number: (8 digit code) * Sample Processing Date: MM/DD/YY

BOTTLE LABEL
* EPA Composite Sample Number: (8 digit code) * Station Location: * Sample Processing Date: MM/DD/YY * Laboratory Samplers Initials: * Type of Sample: 1. Whole body, 2. Fillet with skin 3. Fillet without skin, 4. Eggs

Each sample container should be labeled before filling the bottles with tissue.

To ensure that the bottle labels are attached firmly and will not come off after the bottles are filled with tissue and frozen, the laboratory must wrap an extra layer of clear strapping tape around the bottle completely sticking the tape to itself. As mentioned before, the bottles should not be filled to the rim in order to leave room for some expansion of the tissue when freezing.

8.6 DOCUMENTATION FOR SAMPLE HOMOGENIZATION, ALIQUOT PREPARATION, AND DISTRIBUTION OF ALIQUOTS

8.6.1 Homogenization

The relevant portions of the Sample Processing Record discussed in Section 6.2 and included in Attachment 9 will be completed by the personnel at the subcontract laboratory responsible for homogenization. Each record should be signed and dated upon completion. Copies of this Record will be forwarded to the EPA Work Assignment Manager within 7 calendar days after each batch of samples has been prepared for analyses. In addition, the laboratory will prepare a

narrated video tape showing the procedures and equipment used during each stage of the initial sample processing, including all steps in grinding, mixing and homogenizing, and in cleaning of all equipment. A copy of this video will be sent to the EPA Project Manager after the first batch of samples has been prepared for analysis.

8.6.2 Preparation of Sample Aliquots

The laboratory must maintain accurate records when samples aliquots are prepared for analysis. The Sample Aliquot Record (included as Attachment 12) must be completed by the subcontract laboratory. The Composite Sample ID used on the Sample Aliquot Form should be the one assigned by EPA on the Sample Processing Record. This Sample Aliquot Record should be used to record the total composite homogenate weight for each composite sample and the total number of bottles filled. This record should be signed and dated.

8.6.3 Sample Aliquot Transfer

Laboratory personnel at the analytical laboratories (subcontract laboratory and EPA lab) will be responsible for the care and custody of sample aliquots from the time they are received until the samples are depleted or disposed of. Well documented chain-of-custody procedures must be in place at the laboratory and should include a COC form which must be signed and the date and time noted each time the samples change hands. For sample aliquots being measured by the subcontract laboratory, COC records must be available for review by EPA. For sample aliquots sent to the EPA Laboratory for analysis or archiving, the original field COC Form(s) corresponding to the samples being sent should be signed, enclosed in plastic, and taped to the inside lid of the cooler in which the samples are sent and COC seals applied to the shipping container. All samples/sample aliquots will be shipped on dry ice using the procedures written in Section 7.0. The subcontract laboratory must coordinate with the FOM or other designated staff at the EPA laboratory when samples are to be sent to EPA. **Samples should be sent to the EPA laboratory Monday through Thursday only since the lab is not open on weekends.**

Upon receipt by the EPA laboratory, the sample receipt and chain-of-custody procedures listed in Attachment 11 of this QAPP should be followed.

In addition to the documentation (sample tracking record) required by Attachment 11, the FOM will communicate with the EPA laboratory after they have received the samples to ensure that sample integrity was maintained during shipping. The following information will be communicated to the FOM by telephone or FAX within 24 hours after samples are received: (1) condition of the samples upon arrival at the laboratory (e.g. to ensure sample degradation has not occurred during shipment); (2) time delays (e.g., not arriving the next day); (3) condition of chain-of-custody seals.

The unused portion of the sample will be retained by the analytical laboratories until data validation is completed for the analyses and the Project Manager determines that the DQOs for the samples held at the laboratories have been met without additional analyses. Once the Project Manager has made that determination, the laboratories may dispose of the archived material.

9.0 LABORATORY ANALYSES

As previously discussed, the analysis of polychlorinated dibenzo-p-dioxins and polychlorinated dibenzo-p-furans isomers (PCDDs/PCDFs), percent lipids, and toxic, dioxin-like, PCB congeners will be conducted by a laboratory which is subcontracted by the primary Contractor, Tetra Tech. Pat Cirone will be the Work Assignment Manager (WAM) for this part of the project. The remaining analyses will be measured by the EPA Region 10 laboratory at Manchester, WA.

Laboratory analytical protocols specified for this project are referenced in Table 1 and in the specifications below. Each analytical laboratory which measures project samples will group analytical reports into Sample Delivery Groups (SDGs) as designated by the FOM. SDGs will usually be groups of samples of 20 or less samples. The FOM will designate SDG sizes of 20 samples whenever field conditions permit such a size designation.

Each analytical laboratory which measures project samples will use the following procedure prior to removing a ground sample from a sample bottle for analysis of target compounds:

- Place sample container containing ground fish tissue/eggs in a 34°F to 40°F refrigerator 24 hours prior to removing sample.
- Remove sample bottle from the refrigerator and place on the lab bench at room temperature until all ice crystals in the sample bottle have melted.
- Hand stir the thawed tissue vigorously with a 1/4 inch solid glass rod for 1 minute.
- Immediately remove sample containing tissue and liquid from sample bottle for weighing and laboratory analysis.
- Fill out a Corrective Action Form (see Attachment 18) if any sample bottles contain either chunks of fish tissue or pieces of fish skin. A copy of this Corrective Action Form must be sent to the Project Manager and the Project QA Manager.

9.1 TARGET ANALYTES

For the measurement of PCDDs/PCDFs, target isomers are listed in EPA Method 1613B and in Table 2. Table 3 lists the PCB toxic, dioxin-like, congeners which will be measured using Method 1668.

The EPA Region 10 Laboratory will measure the classes of organics and inorganics listed in Table 1. Target compounds for each class of compounds are listed in Tables 4, 5, 6, 11, and 12. For this project which requires the measurement of pesticide and semi-volatile (SV) organics in fish tissue, it has been difficult to specify the list of target compounds in Tables 4 and 5, because some project samples such as Pacific Lamprey are expected to be composed of 25% by wet weight of lipid compounds. These naturally occurring lipids and fatty acids must be removed from all sample extracts before organic target compounds can be measured. Extract cleanup procedures such as the use of Florisil and silica gel are expected to remove some target compounds listed in Tables 4 and 5. Project quality control measurements for the recovery of laboratory matrix spiked target compounds will provide critical information on the loss of target compounds due to the required use of lipid removing cleanup procedures.

9.2 ANALYTICAL METHODOLOGY

9.2.1 PCDDs/PCDFs

Tetra Tech will be responsible for subcontracting to an analytical laboratory which will be responsible for analysis of PCDDs/PCDFs isomers and toxic, dioxin-like, PCBs according to specifications stated in this QAPP and the following document which is included as Attachment 13:

> EPA Region 10 Statement of Work (Revision 2.1, 6/6/96) For the Measurement of 17 Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzo-p-furans (PCDDs/PCDFs) In Fish Tissue By High Resolution GC/High Resolution Mass Spectrometry Using Method 1613B.

The above Statement of Work (SOW) provides QAPP specifications for the subcontract laboratory in order to permit the measurement of PCDDs/PCDFs in the presence of expected chlorinated chemical interferences, and to provide documented data which will permit EPA to validated PCDD/PCDF data according to the following data validation guidelines (included as Attachment 14):

> EPA Region 10 SOP For the Validation of Polychlorinated Dibenzofuran (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data, Revision 1.4, December 7, 1995.

Tetra Tech shall be responsible for determining if the subcontract laboratory has a QA Program which will support the QA and technical requirements of the QAPP and the analytical

Table 11. Chlorinated Pesticide/Aroclor Target Compound List

Target Compound	CAS Number	Risk Level [1] ug/Kg	Quantitation Limit ug/Kg [2]
4,4'-DDE	72-55-9	1.1	3.3
4,4'-DDT	50-29-3	1.1	3.3
4,4'-DDD	72-54-8	1.5	3.3
Aldrin	309-00-2	0.021	17
alpha-BHC	319-84-6	0.057	1.7
alpha-Chlordane	5103-71-9	0.28	1.7
beta-BHC	319-85-7	0.20	1.7
delta-BHC	319-86-8	NC	1.7
Dieldrin	60-57-1	0.023	33
Endosulfan I	959-98-8	0.90	17
Endosulfan sulfate	1031-07-8	NC	33
Endosulfan II	33213-65-9	0.90	33
Endrin	72-20-8	5.4	33
Endrin aldehyde	7421-36-3	NC	33
Endrin ketone	53494-70-5	NC	33
gamma-Chlordane	5103-74-2	0.28	1.7
gamma-BHC(Lindane)	58-89-9	0.28	1.7
Heptachlor epoxide	1024-57-3	0.040	1.7
Heptachlor	76-44-8	0.080	1.7
Hexachlorobenzene	118-74-1	0.23	1.7
Methoxychlor	72-43-5	89.5	170
Pentachloroanisole	1825214	NC	1.7
Toxaphene	8001-35-2	0.33	170.0
Aroclor-1016	12674-11-2	0.047	33.0
Aroclor-1221	11104-28-2	0.047	67.0
Aroclor-1232	11141-16-5	0.047	33.0
Aroclor-1242	53469-21-9	0.047	33.0
Aroclor-1248	12672-29-6	0.047	33.0
Aroclor-1254	11097-69-1	0.047	33.0
Aroclor-1260	11096-82-5	0.047	33.0

Table 12. AED/Pesticide Target Compound List	
Target Compound[1]	**CAS Number**
Abate (Temephos)[3]	3383-96-8
Alachlor	15972-60-8
Ametryn	834-12-8
Atraton	1610-17-9
Atrazine	1912-24-9
Azinphos Ethyl (Ethyl guhion)	642-71-9
Azinphos methyl (Guthion)	86-50-0
Benfluralin	1861-40-1
Bromacil	314-40-9
Butachlor	23184-66-9
Butylate	2008-41-5
Captafol [3]	2425-06-1
Carbophenothion	786-19-6
Carboxin [3]	5234-68-5
Chlorpropham	101-21-3
Chlorpyrifos	5598-13-0
Chlorthalonil (Daconil)	1897-45-6
Coumaphos	56-72-4
Cyanazine [3]	21725-46-2
Cycloate	1134-23-2
DCPA (Dacthal)	2136-79-0
DEF (Butifos)	78-48-8
Diallate	2303-16-4
Diazinon	333-41-5
Dichlobenil (Casoron)	1194-65-6
Dichlorvos (DDVP)	62-73-7
Dimethoate [3]	60-51-5
Dioxathion [3]	78-34-2
Diphenamid	957-51-7

Table 12. AED/Pesticide Target Compound List	
Target Compound[1]	**CAS Number**
Disulfoton (Disyston)	298-04-4
EPN	2104-64-5
Eptam (EPTC)	759-94-4
Ethalfluralin (Sonalan)	55283-68-6
Ethion	563-12-2
Ethoprop	13194-48-4
Fenamiphos	22224-92-6
Fenarimol	60168-88-9
Fenitrothion	122-14-5
Fensulfothion	115-90-2
Fenthion	55-38-9
Fluridone [3]	59756-60-4
Fonofos	944-22-9
Gardona (Tetrachlovinphos)	961-11-5
Imidan (Phosmet)	732-11-6
Malathion	121-75-5
Merphos	150-50-5
Metalaxyl	57837-19-1
Methyl chlorpyrifos	5598-13-0
Methyl parathion	298-00-0
Metolachlor	51218-45-2
Metribuzin	21087-64-9
Mevinphos	7786-34-7
MGK-264	113-48-4
Mirex	2385-85-5
Molinate	2212-67-1
Napropamide	15299-99-7
Norflurazon [3]	27314-13-2
Oxyfluorfen	42874-03-3
Parathion	56-38-2

Table 12. AED/Pesticide Target Compound List	
Target Compound[1]	**CAS Number**
Pebulate	1114-71-2
Pendimethalin	40487-42-1
Phorate	298-02-2
Phosphamidan [3]	297-99-4
Profluralin	26399-36-0
Prometon (Pramitol 5p)	1610-18-0
Prometryn	7287-19-6
Pronamide (Kerb)	23950-58-5
Propachlor (Ramrod)	1918-16-7
Propargite (S-181)	2312-35-8
Propazine	139-40-2
Propetamidophos	31218-83-4
Ronnel	299-84-3
Simazine	122-34-9
Sulfotepp	3689-24-5
Sulprofos (Bolstar)	35400-43-2
Tebuthiuron	34014-18-1
Terbacil	5902-51-2
Terbutryn (Igran)	886-50-0
Triademefon	43121-43-3
Triallate	2303-17-5
Trifluralin (Treflan)	1582-09-8
Vernolate [3]	1929-77-7

[1] -- Some compounds in this target compound list are expected to be lost during extract clean-up procedures.
[2] -- Quantitation limits are for fish tissue on a wet weight basis.
[3] -- It is uncertain if this target compound will survive clean-up procedures.

Statement of Work, above. In order for Tetra Tech to determine if the subcontract laboratory has an adequate QA Program, Tetra Tech shall review and comment upon the following documents from each subcontract laboratory source which submits a bid proposal for this Task:

1. Results of the measurement of EPA Water Supply performance evaluation (PE) samples over the past 2 years for the measurement of 2,3,7,8-TCDD.

2. Laboratory Quality Assurance Plan.

3. Standard Operating Procedures (SOPs) for the measurement of fish tissue samples using Method 1613B and for the measurement of fish tissue samples which meet the data quality objectives which are specified in the QAPP and the Laboratory SOW for the project. These SOPs must document the procedure that the laboratory will use to obtain an initial calibration of 2,3,7,8-TCDD and 2,3,7,8-TCDF between 0.1 ng/ml and 200 ng/ml.

The above three types of documents will be reviewed by Tetra Tech to determine if the subcontract laboratory(s) has a comprehensive QA program and the facilities, staff, and experience to meet the QA requirements and Data Quality Objectives of the QAPP.

The quantitation limits specified in Table 2 for the measurement of 2,3,7,8-TCDD and 2,3,7,8-TCDF require that the subcontract laboratory achieve a Minimum (Quantitation) Limit (ML) of 0.2 ng/Kg (wet weight) for isomers 2,3,7,8-TCDD and 2,3,7,8-TCDF. This lower ML shall be achieved by the use of a low initial calibration point of 0.1 ng/ml and an ultra-low sensitivity HRMS system.

Tetra Tech will provide a data package which addresses all the data assessment requirements of Method 1613B and the EPA data validation SOP.

9.2.2 Toxic, Dioxin-Like, PCBs

Tetra Tech (Contractor) will be responsible for subcontracting to an analytical laboratory which will be responsible for analyses of toxic, dioxin-like, PCB congeners listed in Table 3. These analyses shall be done according to the specifications stated in this QAPP and the following document (included as Attachment 15):

Draft Method 1668 For the Measurement of Toxic PCB Congeners By Isotope Dilution HRGC/HRMS, October 4, 1995 Draft Revision.

All the required standards and isotopes to measure samples using Method 1668 are currently commercially available. Similar to the measurement of PCDDs and PCDFs, the toxic, dioxin-like, PCBs will be validated by EPA Region 10 according to a validation guidelines (SOP) developed by Region 10 (see Attachment 16).

9.2.3 Pesticides/Aroclors

The Region 10 Laboratory will measure chlorinated pesticides/PCB mixtures (as Aroclors), other pesticides including nitrogen and organo-phosphorous pesticides by AED (Method 8085), neutral SVs, chlorinated phenolics and inorganic target compounds listed in Table 1 and Tables 4, 5, 6, 11, and 12 using EPA Laboratory SOPs.

The homogenized tissue samples will be extracted utilizing the Soxhlet technique as described in the "National Study of Chemical Residues in Fish", EPA 823-R-92-008a, September 1992. This extraction procedure is analogous to SW-846 Method 3540B. The extract volume will be split with one third of the volume used for Semivolatiles (Tables 4 and 5) and two thirds of the volume used for pesticides (Tables 11 and 12). Extracts for pesticides/Aroclors listed in Tables 11 and 12 will require Florisil cleanup (SW-846 Method 3620A), generating two fractions, 0% and 100%.

The 0% fraction will be treated with concentrated sulfuric acid (SW-846 Method 3665), to remove any GC/ECD interferences and analyzed for PCBs, DDE, Heptachlor and Aldrin. These compounds are not acid labile. The 100% fraction will be cleaned up using an acetonitrile partitioning step to remove lipids. After removal of lipids, the extract will be split. The split for AED analysis will not require additional clean up except for possible sulfur removal with elemental mercury, SW-846 Method 3660A. The split for GC/ECD analysis for the remaining Chlorinated Pesticides will be partitioned again using Florisil chromatography, SW-846 Method 3620A, generating a 6% fraction, a 15% fraction and a 50% fraction. All three fractions will receive mercury treatment to remove elemental sulfur. A portion of the 6% fraction will be treated with concentrated sulfuric acid. The 6% fractions, 15% fraction, and 50% fraction will be analyzed primarily by GC/ECD for Chlorinated Pesticides.

All project samples will be measured for the chlorinated pesticides and Aroclors listed in Table 11. Samples will be measured in batches of approximately 20 samples. Each batch will also consist of 2 method blanks and 4 MS/MSD samples.

The split for AED analysis and all Pesticide/Aroclor extracts will be saved for future potential AED analysis. These extracts will be sealed in containers and kept in the freezer. Depending on the results of the chlorinated pesticide/Aroclor analysis and the location of project samples, a subset of the project samples will be analyzed by atomic emmision detector (AED). The subset will include a few samples with low concentrations as well as samples with high chlorinated pesticide/Aroclor concentrations. The Project QAM with concurrence of the Project Manager will designate between 60 to 120 project samples which will be measured for the additional pesticides listed in Table 12 using AED Draft Method 8085. Quantitation limits for AED target compounds listed in table 12 are unknown, because most of these compounds have not been previously measured in the fish matrix using Method 8085.

The spiking protocol for chlorinated pesticides/Aroclors will be as follows:

All extraction sets for chlorinated pesticides and PCBs will receive both the chlorinated-pesticide mix of 19 pesticides and the PCB mix of Aroclors 1242 and 1260 for Table 11 target compounds. In addition the organochlorine spiking mixes #2 and #3, as well as the

organophosphate mixes #1, #2, and #3 and nitrogen-containing pesticide spiking mixes #1, #2, and #3 for the 8085 method will be added on a rotating basis:

- Set 1 (first batch of 20 samples)
  chlorinated pesticide mix #1 and PCB 1242/1260 mix
  O-pesticide/ N-pesticide mix #1

- Set 2 (next set of 20 samples)
  chlorinated pesticide mix #1 and PCB 1242/1260 mix
  Cl/ O-pest/ N-pest mix #2

- Set 3 (next set of 20 samples)
  chlorinated pesticide mix #1 and PCB 1242/1260 mix
  Cl/ O-pest/ N-pest mix #3

Each set of 20 samples will have different target compounds or the same target compound at different spiking concentration levels.

After the third set, the cycle goes back to Set 1 protocol for Cl/ O-pest/ N-pest.

9.2.5 Neutral Semivolatiles

Neutral SV target compounds are listed in Table 4. Extracts will be cleaned up using gel permeation chromatography (GPC) followed by silica gel column chromatography to isolate a neutral fraction containing PAHs and compounds. Target compounds will be measured using HRGC/LRMS/SIM in order to achieve the quantitation limits listed in Table 4.

9.2.6 Chlorinated Phenolics

This group of phenolics listed in Table 5 will be extracted, derivatized by acetylation, and analyzed using a modification to the procedure described in draft Method 1653.

A synopsis of the analytical procedure for the analysis of the chlorinated phenolics is as follows. A portion of the hexane extract prepared from the fish tissue is added to a stir-bar extraction vessel containing one liter of potassium carbonate buffer. Internal standard and surrogate are added and the mixture stirred. Acetic anhydride and hexane are added and the mixture stirred to simultaneously derivatize and extract the derivatives. If necessary, extracts will be cleaned up by either silica gel or alumina chromatography. Additional details are described in Manchester SOP 730016_7/93.

9.2.7 Metals

Cold mercury measurements of project tissue samples are described in EPA Region 10 SOP Automated Mercury Analysis of Tissue Samples by Cold Vapor Atomic Absorption (CVAA) Using Leeman Labs' PS200 or PS200ii, Revision 11/27/96.

Mercury measurements on project tissue samples are described in EPA Region 10 SOP "Automated Mercury Analysis of Tissue Samples by Cold Vapor Atomic Absorption (CVAA) Using Leeman Labs' PS200 or PS200ii, Revision 11/27/96."

The remainder of metals listed in Table 6 will be digested using a modified Method 200.3 and measured by ICP/MS using Method 200.8. A freeze-dried fish reference sample will be measured with each of project samples which are digested.

A summary of the procedure is as follows.

Samples are digested in batches of 20, with duplicate, spike/spike duplicate, spike and method blank and reference material.

Five gram subsamples of homogenized fish tissue are transferred to 250 mL pre-cleaned Teflon beakers. The tissues are digested in a Class 100 hood as specified in the EPA method 200.3. The addition of hydrochloric acid is omitted to avoid interferences produced by the chloride ion during ICP/MS analysis.

Hydrogen peroxide is added to a maximum of six mL and the multi element spike is added to give a concentration of 30 ug/L in the analytical solution for each element.

After a period of cooling, the samples are transferred to 125 mL polyethylene pre-cleaned bottles and diluted with ASTM type I water to 100 mL. The samples are then left to settle any insoluble material and then diluted five times with deionized water.

The reference material used is DORM-2, freeze-dried dogfish muscle and liver, from the National Research Council Canada. The amount of DORM-2 digested is 0.5 grams.

The samples are analyzed as soon as possible after digestion by ICP/MS using the EPA method 200.8. Samples are measured against a linear,four point calibration curve forced through the origin,and results are reported in mg/Kg wet weight.

The reference material is only being analyzed as a measure of precision throughout this long term project. DORM-2 is a different matrix than and no representative of the digested frozen tissue. A frozen tissue reference sample does not exist and this is the next best alternative.

9.3 CALIBRATION PROCEDURES AND FREQUENCY

Calibration and frequency of calibration of laboratory instruments shall be according to the requirements of each method of analysis. These requirements are listed in the methods (Attachments 13 and 15 for PCDD/PCDFs and dioxin-like PCBs, respectively, and in Table 1 for

other analytes) for each class of chemicals to be analyzed. Each laboratory shall have a Standard Operating Procedure (SOP) which describes how each target compound will be measured. The EPA Region 10 Statement of Work (Revision 2.0, 12\5\95) For the Measurement of 17 Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzo-p-furans (PCDDs/PCDFs) In Fish Tissue By High Resolution GC/High Resolution Mass Spectrometry Using Method 1613B, QAPP Attachment 13, sets specifications for calibration of 2,3,7,8-TCDF on a second confirmation column. Tetra Tech shall provide copies the subcontract laboratory's analytical SOPs to EPA.

9.4 LABORATORY QC PROCEDURES

Quality Control procedures specified in the QAPP and in the methods listed in Table 1 shall be followed and documented by each laboratory. In addition, Section 3.0 of the QAPP specifies that all quality control requirements of each method which is referenced in Table 1 shall be obtained and reported by each analytical laboratory, which includes QC requirements for surrogate compounds, internal standards, recovery standards, matrix spike compounds, calibrations, and method blanks.

10.0 ANALYTICAL DATA VALIDATION AND REVIEW

This section describes data validation, which is the process of technically reviewing analytical data using written data validation protocols, and qualifying measurement results using data qualifiers. The primary objective of data validation is to determine if project data meets the data quality objectives which are specified in the QAPP. After the data validation process is completed, data qualifiers are appended to measurement values by the data validation chemist. Final useability of qualified and validated data is determined by data users such as the Project Manager, CRITFC members, and local community members.

10.1 DATA VALIDATION

Data validation of PCDD/PCDF and toxic, dioxin-like, PCB data will be conducted by EPA Region 10. The following written protocols will be used for PCDD/PCDF and toxic, dioxin-like, PCB data:

> EPA Region 10 SOP For the Validation of Polychlorinated Dibenzofuran (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data, Revision 1.4, December 7, 1995. (Attachment 14)
>
> EPA Region 10 SOP For the Validation of Method 1668 Toxic, Dioxin-Like, PCB Data, Revision 1.0, December 8, 1995, (Attachment 16).

The Project QA Manager will provide data validation reports for PCDD/PCDF and toxic, dioxin-like, PCB data to the Project Manager.

EPA Region 10 Laboratory staff will perform a standard laboratory data validation of Region 10 Laboratory data using the following guidelines:

> EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (PB-94-963502)(5)
>
> EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (PB-94-963501)(6)

The Project QA Manager will provide an assessment and evaluation of data validation reports. Criteria for the assessment and evaluation of data validation reports will be based upon the validation criteria which is specified in the above data validation SOPs and EPA data validation guidelines. Data outliers such as data qualified with "J" and "R" flags will be documented in data

validation reports to the Project Manager. Data validation guidelines require that measurement values below the quantitation limit be qualified as an estimated value. Data users such as risk assessors will determine the useability of such estimated values. If resources are available, the Work Assignment Manager may elect to have "R" qualified samples reanalyzed using archived samples.

10.2 DATA ASSESSMENT PROCEDURES

Following the data validation process, validated data will be assessed by the Project Manager to determine if the data meets the DQOs of the project plan. This assessment of validated data will be reported in the Final Report for the project.

11.0 PERFORMANCE AND SYSTEM AUDITS

Performance and systems audits for field work, filleting, homogenization and analyses will be conducted according to the following schedule:

11.1 AUDITS RELATED TO SAMPLE COLLECTION AND SAMPLE FILLETING

The Project QA Manager or his designee may conduct an on-site systems audit during sample collection and filleting field activities. An oral report of the results of any audits will be made to the Project Manager within 2 days of completion of each audit. A written report will be submitted within two weeks of each field audit.

11.2 AUDITS RELATED TO COMPOSITING AND HOMOGENIZATION OF FISH TISSUE

The FOM at Region 10 Office may conduct an on-site Technical Systems Audit of the subcontract laboratory which is responsible for grinding and compositing project fish samples. If this audit is conducted, written results will be submitted to the Project QA Manager and to the Project Manager within two weeks of the date of the audit.

In addition, the Region 10 FOM or his designate will inspect and document the nature of samples that are composited and ground by the subcontract laboratory when these samples are received by the Region 10 laboratory. The results of this audit and these inspections will be reported orally to the Project QA Manager and the Project Manager within two days of the audit and inspections. A written report will be submitted within two weeks of each inspection.

11.3 AUDITS RELATED TO SAMPLE ANALYSES

Tetra Tech shall conduct a Technical Systems Audit of the analytical subcontract laboratory. Tetra Tech shall develop, with EPA review and approval, an audit checklist that will be used in auditing the subcontract laboratory. The checklist will include QAPP requirements, method requirements, and any additional requirements established by Tetra Tech's work assignment. The subcontract laboratory shall prepare a report for the WAM (EPA Project Manager), based on the audit, which shall identify any instances in which the analytical laboratory work does not meet the requirements specified in the QAPP or Tetra Tech's Work Assignment. Tetra Tech shall provide the WAM with a advance notice of the audit and shall afford EPA and its technical advisors on the project the opportunity to participate in the audit as observers.

The subcontract laboratory which is responsible for measuring PCDDs/PCDFs and toxic, dioxin-like, PCBs will procure and measure PE samples EDF-2524, EDF-2525, and EDF-2526 when the first Sample Delivery Group is measured using Methods 1613B and 1668. Tetra Tech will designate a second SDG during the latter phase of the project for the subcontract laboratory to measure PE samples EDF-2524, EDF-2525, and EDF-2526. The results of the measurement of PCDDs/PCDFs and toxic, dioxin-like, PCBs in these PE samples will be evaluated by the Project QA Manager within 14 days of receipt of data from Tetra Tech using the data validation report and the mean value and confidence intervals (at the 95% confidence level) of the interlaboratory study. The evaluation of PE measurement results will be summarized in the data validation report which is sent to the Project Manager. The Project Manager (WAM) will require corrective actions of Tetra Tech if the subcontract laboratory submits PE sample results which are determined by EPA to be outside the 95% confidence level of the interlaboratory study.

Other types of Performance or Systems Audits of field or laboratory activities may be scheduled by the Project Manager.

12.0 PREVENTATIVE MAINTENANCE

Preventive maintenance will take two forms: 1) implementing a schedule of preventive maintenance activities to minimize downtime and ensure accuracy of measurement systems, and 2) ensuring stock of critical spare parts and backup systems and equipment. The preventive maintenance approach for specific pieces of equipment used in sampling, monitoring, and documentation will follow manufacturer specifications and method requirements. Performance of these maintenance procedures will be documented in field logbooks and laboratory notebooks.

All laboratories will have service contracts in place for measurement systems which are used to measure project samples. The EPA Manchester Laboratory and Tetra Tech may be required by the Project Manager (WAM) to provide documentation that each laboratory which measures project samples have a preventive maintenance program and service contracts in place for measurement systems which are used to measure project samples.

Each laboratory will follow the preventive maintenance procedures specified in approved SOPs.

13.0 CORRECTIVE ACTIONS

Corrective actions taken during the sample collection and analysis phase of the project fall into two categories: 1) analytical or equipment malfunctions which could affect the ability of project staff or Tetra Tech to meet the stated requirements of the QAPP and 2) nonconformance or noncompliance with QA requirements set forth for the project.

Attached to the QAPP are a SAMPLE ALTERATION FORM (Attachment 17) and CORRECTIVE ACTION FORM (Attachment 18). These forms will be used to report problems that occur in the field, such as changes in the location or nature of samples collected, and in the EPA, Region 10 laboratory. The subcontract laboratory can use these forms or equivalent ones to report to Tetra Tech. Tetra Tech will forward a copy of these forms to the WAM and the Project QA Manager. In addition, each laboratory will provide a Case Narrative with the laboratory Data Report which will specify any problems which occur during the measurement of project samples.

SAMPLE ALTERATION FORMS and CORRECTIVE ACTION FORMS are initiated by any staff member of the Project or any staff member of Tetra Tech or laboratories which process Project samples. All SAMPLE ALTERATION FORMS and CORRECTIVE ACTION FORMS are signed by a Project Manager and the Project QA Manager. A file of all SAMPLE ALTERATION FORMS and CORRECTIVE ACTION FORMS implemented for Project activities will be maintained by the Project QA Manager.

It is the responsibility of the Project QA Manager to ensure that corrective actions are taken and recorded for all problems which are documented by CORRECTIVE ACTION FORMS, by field or laboratory audits, or by data validation evaluations. The Project QA Manager will document and report all of the above project problems to the Project Team Leader and the Project Manager. The Project Manager will initiate corrective actions in the event of lost samples or unusable project samples.

14.0 REPORTING REQUIREMENTS AND DELIVERABLES

This section briefly describes the deliverables and reporting requirements that are expected for this project. Deliverables are required from both Tetra Tech and the subcontract laboratory, as well as from the EPA, Region 10, field staff, laboratory, and QA Unit. Reporting requirements apply to Tetra Tech only. Contractor deliverables and reporting requirements and their due dates are described in detail in the Work Assignment for the EPA Contractor for this project and are summarized below.

14.1 FIELD WORK

Samples (whole fish and fillets, and eggs) will be collected by EPA with help from CRITFC. Fish tissue samples (with or without filleting) will be sent on dry ice via Federal Express to the processing laboratory with the appropriate documentation (i.e., SI/COC Tags, Sample Processing Records, and Chain-of-Custody Forms). The Field Record Form and notebook will be retained by the FOM.

14.2 FISH PROCESSING

Within 7 calendar days of receipt of fish samples, the subcontract laboratory will process the fish samples and distribute the sample aliquots for analysis to both the subcontract laboratory and the EPA, Region 10 laboratory. Deliverables include:

(1) the sample aliquot jars and the reusable shipping containers to the EPA laboratory,

(2) documentation (the COC Forms to the EPA laboratory with the sample aliquots; the Sample Processing Records, Sample Aliquot Records, and videotapes documenting sample processing) to the WAM.

As discussed above in Section 11.2, the FOM may conduct an on-site audit of the subcontract laboratory to ensure that fish processing is completed according to specifications of the QAPP. In addition, each batch of processed fish and egg samples will be inspected as they arrive at the Region 10 laboratory for analysis. The results of the fish processing laboratory audit, if conducted, and of the inspection of the processing of fish samples at the Region 10 laboratory will be documented and copies will be provided to the Project Manager (WAM) and the Project QA Manager within two weeks of their completion. Any problems noted with fish processing will be reported orally to the Project Manager and the QA Manager within 2 days.

14.3 LABORATORY ANALYSES

Detailed communication logs concerning this project and the preparation and analysis of project samples shall be maintained by Tetra Tech and subcontract laboratory. Copies of these logs shall be submitted to the WAM and Project QA Manager, in addition to any corrective action and sample alteration forms.

Within 35 days of verified time of shipment of homogenized and composited samples from the subcontract laboratory to the EPA Manchester Laboratory (subcontract laboratory must homogenize and composite project samples within 7 days of verified time of sample receipt), analytical results for Methods 1613B and Method 1668 shall be reported to Tetra Tech. Tetra Tech must submit data packages of analytical results from the subcontract laboratory to the WAM within 7 days of receipt from the subcontract laboratory. The Project QA Manager will perform the data validation review.

For PCDDs/PCDFs, a detailed description of required data documentation is given in the analytical SOW (Attachment 13) for the subcontract laboratory. In general, the subcontract laboratory shall provide all original data to document that all requirements of Method 1613B have been met. All raw data shall be submitted, along with example calculations, such that an independent data reviewer may recreate the calculations reported by the laboratory. In order to check for polychlorinated diphenyl ether (PCDPE) interferences, the subcontract laboratory shall submit simultaneous offset display of single ion chromatogram for each GC column for analyte peaks and for PCDPE peaks which may co-elute with native target compounds, according to the specifications of the PCDD/PCDF SOW (Attachment 13). Similar type documentation will be submitted for the analysis of toxic (dioxin-like) PCB congeners as discussed in Attachment 15.

The EPA Region 10 laboratory at Manchester will provide both analytical data from the analyses of other organics and all inorganics, and provide data validation reports. Data analytical reports of Manchester Laboratory data will be due within 45 days from verified time of sample receipt. Validation reports from the Manchester Laboratory will be delivered to the Project Manager within 75 days from verified time of sample receipt.

As described in Section 11.3, an on-site Technical Systems Audit of the subcontract laboratory will be conducted by Tetra Tech. EPA will be afforded the opportunity to observe the audit process. A checklist to be used during the audit will be developed by Tetra Tech with review and approval from EPA. Tetra Tech and any EPA staff participating in the audit will provide separate verbal observation reports to the WAM within 2 working days of the audit. A written report of audit results will be prepared by Tetra Tech and submitted to EPA WAM within 14 working days of the audit. If the written audit report indicates conditions at the laboratory that may compromise the project or DQOs of the QAPP, the WAM will immediately contact Tetra Tech to request corrective actions.

14.4 DATA SUMMARY FINAL REPORT AND DATABASE UPDATE

Data Summary Report - The analytical data generated and validated by the EPA Laboratory will be sent to the Project QA Manager to determine if the data validation meets the DQOs described in the QAPP. The analytical data generated by the subcontract laboratory will be validated by the Project QA Manager. The validated data from both laboratories will then be sent to Tetra Tech who will compile it into a Final Data Summary Report. A draft summary data report will first be prepared and sent to the WAM for review. Within 2 weeks of receiving comments from EPA, Tetra Tech will submit the final summary data report. This report will summarize all of the analytical data for each target species at each sampling site as well as additional data specified in Tetra Tech's Work Assignment. The format and schedule for preparation of the Data Summary Report are described in Tetra Tech's Work Assignment.

Columbia River Contaminant Database Update - Tetra Tech will enter the validated analytical data from all analyses from this project into the Columbia River Contaminant Database and check the data for errors after data entry is completed. The schedule and deliverables for completion of the database update are described in Tetra Tech's Work Assignment.

15.0 QA REPORTS TO MANAGEMENT

The Region 10 Quality Assurance Unit will provide assistance to the Project Manager in reviewing the QAPP and in performing audits of selected project activities when requested by the Project Manager.

The results of audits specified in Section 10, above, will be submitted within 2 weeks of completion of each requested audit. Problems noted during the audits will be reported orally within 2 working days.

The Project QA Manager will submit written QA reports to the Project Manager when requested. These reports may include the following:

- Project status reports.
- Results of performance and systems audits
- Summary of significant QA problems and the corrective actions taken to correct these problems.
- Requests for changes or modifications to the QAPP.
- Results of data quality assessments of project data.
- Data validation reports for PCDD/PCDF and toxic, dioxin- like, PCBs data.

16.0 REFERENCES

(1) A Fish Consumption Survey of the Umatilla, Nez Perce, Yakama, and Warm Springs Tribes of the Columbia River Basin. CRITFC Technical Report No. 94-3. Portland, Oregon.

(2) Assessment of Chemical Contaminants in Fish Consumed by Four Native American Tribes in the Columbia River Basin - Draft Scoping Document, prepared for the U.S. EPA by Tetra Tech, September 30, 1994.

(3) Assessment of Chemical Contaminants in Fish Consumed by Four Native American Tribes in the Columbia River Basin - Final Draft Study Design, prepared for the U.S. EPA by Tetra Tech, December 2, 1994.

(4) Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume I: Fish Sampling and Analysis (U.S. EPA 1993b).

(5) EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (PB-94-963502).

(6) EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (PB-94-963501).

(7) Persistence of the DDT Pesticide in the Yakima River Basin Washington, U.S. Geological Survey, Circular 1090, 1993.

ATTACHMENTS

Attachment 1. Cooperative Agreement Between the Columbia River Inter-Tribal Fish Commission and the U.S. EPA

Attachment 2. EPA, Region 10, Boat Operating Policy

Attachment 3. Electrofishing Safety Procedures

Attachment 4. Field Record Form

Attachment 5. Sample Identification/Chain of Custody Tag

Attachment 6. EPA, Region 10, Chain of Custody Form

Attachment 7. Sections 7.2.1 and 7.2.1.3 of "Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories: Volume 1, Fish Sampling and Analysis"

Attachment 8. Sections 7.2.2.6 and 7.2.2.7 and Figure 7-3 of "Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories: Volume 1, Fish Sampling and Analysis"

Attachment 9. Fish Processing Record

Attachment 10. Custody Seal and Hazardous Substances Label

Attachment 11. Sample Receipt and Chain of Custody

Attachment 12. Sample Aliquot Record

Attachment 13. EPA Region 10 Statement of Work (Revision 2.2, 6/17/96) For the Measurement of 17 Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzo-p-furans (PCDDs/PCDFs) In Fish Tissue By High Resolution GC/High Resolution Mass Spectrometry Using Method 1613B.

Attachment 14. EPA Region 10 SOP For the Validation of Polychlorinated Dibenzofuran (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data, Revision 1.4, December 7, 1995.

Attachment 15. Draft Method 1668 For the Measurement of Toxic PCB Congeners By Isotope Dilution HRGC/HRMS, October 4, 1995 Draft Revision.

Attachment 16. EPA Region 10 SOP For the Validation of Method 1668 Toxic, Dioxin-Like, PCB Data, Revision 1.0, December 8, 1995.

Attachment 17. Sample Alteration Form

Attachment 18. Corrective Action Form

Attachment 19. 1996 Summer Sampling Design For the CRITFC Exposure Study

Attachment 20. Previous 6/11/96 Sampling Design For the CRITFC Exposure Study

Attachment 21. Previous Sampling Map From 6/17/96 QAPP